september, 1961

nlgi spokesman

journal of the national lubricating grease institute

Revised NLGI GLOSSARY—New Additions to Definitions of Terms Relating to the Lubricating Grease Industry

By A. S. ORR

Electron Microscope Examination of Thin Sections of Lubricating Grease Thickeners

By J. W. WILSON, JR.

nlgi spokesman

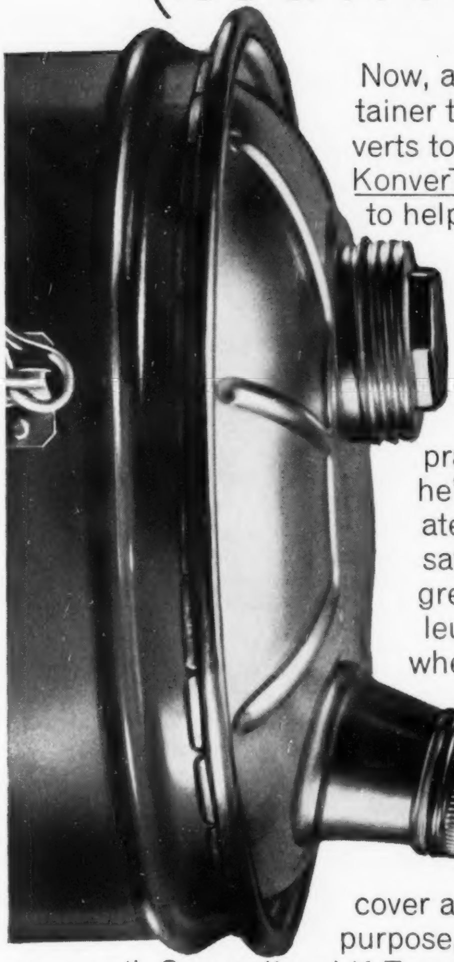
Volume XXV September, 1961 Number 6

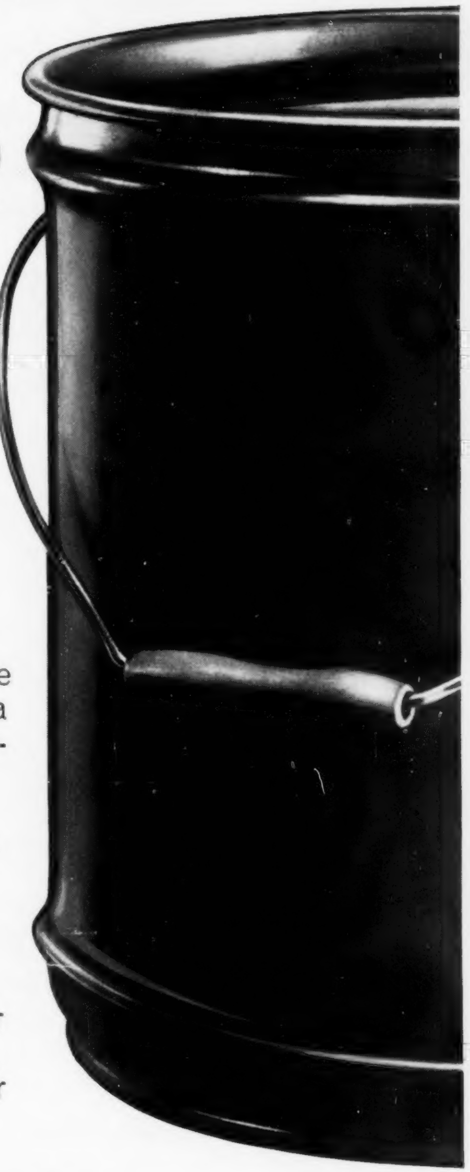
Published Monthly by the National Lubricating Grease Institute, T. W. H. MILLER, Editor; VIRGINIA ALLEN, Assistant Editor, 4638 J. C. Nichols Parkway, Kansas City 12, Missouri. Telephone VAlentine 1-6771. 1 Year Subscription, $5.00. 1 Year Subscription (Foreign), $6.00.

IN THIS ISSUE

THE COVER

HOUSTON's ever changing skyline (note the new building going up on the right) is only one facet of this dynamic, bustling city. With a growth rate predicted to rival that of Los Angeles, this seaport, manufacturing center, and vital petroleum industry headquarters town will be the host city of NLGI's 29th Annual Meeting. There is a myriad of things to do in Houston, the Gulf is just minutes away, and Mexico can be reached in hours. On top of that there is an excellent program as outlined tentatively, beginning on page 158.





NLGI PRESIDENT'S PAGE

By F. R. HART, President

Much to Gain At Annual Meeting

It is almost annual meeting time again and I know you will be interested to learn about the program your Vice-President is preparing. First, the meeting is to be held at the Rice Hotel in Houston, Texas, October 30, 31 and November 1, 1961.

As was the case in 1959 and again in 1960, an Early Bird social will be held on Sunday, October 29, 1961, in the Grand Ball Room, Rice Hotel. Last year, this was an outstanding event and judging from sponsor interest, it will be an even more elaborate social this year. Don't miss this plus value. In addition, preparations have been completed to take the ladies and their escorts on a delightful (no-cost) boat ride. Those who take in this event Tuesday afternoon October 31 will embark at Houston and cruise down the ship channel to San Jacinto Monument and Inn where they will disembark. The return trip to the hotel will be made via bus—and you know how much fun this can be.

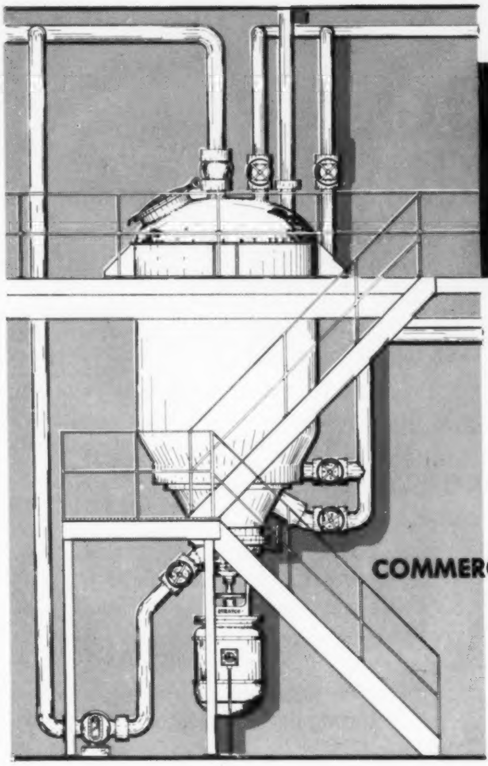
Vice-President Claude Johnson has devoted much time, thought and energy to the program. The subjects he has selected are those that will appeal to the experienced grease maker, yet they recognize the importance of new and specialized lubricants, modern manufacturing plants, equipment and the continuing need for emphasis on marketing opportunities.

Your capable Chairman of the Technical Committee, L. C. Brunstrum, is putting together a joint symposium (NLGI-ASTM) featuring the effect of lubricating oil viscosity on the flow properties and service performance of greases. Five papers will be presented on this subject during a concurrent session.

Of particular interest will be the keynote address to be delivered Monday A.M., October 30. The subject has not been revealed at this writing but I am sure it will be timely, stimulating and provide the enthusiasm so typical of NLGI annual meetings.

Another outstanding contributor is Mr. Hugh Hemmingway of the Pure Oil Company. Many of you will remember Hugh as President of NLGI during 1955 and the outstanding job he did for the Institute. Hugh is well-qualified to talk on empathy between researcher and marketer.

I am looking forward to meeting with you at Houston next October 29th. I hope you can be there to enjoy the business session and the fun.

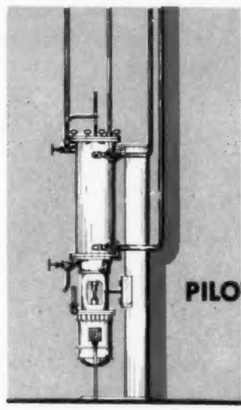


STRATCO

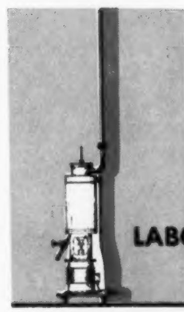
GREASE CONTACTORS

- **Simplify Production**
- **Reduce Costs**
- **Produce More Uniform Greases**

COMMERCIAL



PILOT



LABORATORY

Used in a complete Stratco Installation or as units in existing plants, Stratco Contactors provide continuous or batch mixing with very short time cycles, less soap, less laboratory control.

They replace other less efficient equipment, simplify plant layout, make possible either increased production or fewer man hours of operation.

Available in three types as illustrated so experimental and pilot plant results can be used for the formulation and plant guidance of full scale commercial equipment. Intermediate sizes to meet all requirements. Write for details.

STRATFORD ENGINEERING CORPORATION • PETROLEUM REFINING ENGINEERS

612 West 47th St. Kansas City 12, Mo.

REPRESENTATIVES

D. D. Foster Co., Pittsburgh, Pa.
D. D. Foster Co., S. Charleston, W. Va.

Lester Oberholtz, Los Angeles
Rawson-Houlihan Co., Inc., Houston
Rawson-Houlihan Co., Inc., Beaumont, Texas

The Rawson Co., Inc., Baton Rouge
F. J. McConnell Co., New York

C960

News About NLGI

As We Go to Press . . .

Albert S. Orr

Services for Albert S. Orr, 58, director of product quality coordination, refining department, Gulf Oil corporation, were held Wednesday, August 16. Mr. Orr died August 13 while vacationing at Long Beach, N. J. He was a resident of Mt. Lebanon, Pa.

A native of Texas, Mr. Orr graduated from Texas A&M college in 1924 with a BS degree in chemical engineering, and joined Gulf at its Port Arthur refinery that same year. In 1930, he was transferred to the company's refinery technology laboratory in Philadelphia; and in 1950 was made a director of the laboratory. He was appointed assistant manager, technical division and transferred to the general office, Pittsburgh, in 1951, and named director of product quality coordination in 1960.

Active in industry affairs, Mr. Orr was a member of the American Chemical Society, the Society of Automotive Engineers, the American Society for Testing Materials and the National Lubricating Grease Institute, where he had been a subcommittee chairman on terms and definitions (see page 160).

Change Representatives

Mallinckrodt Chemical Works, St. Louis, Mo., has named John A. Caughlin as Company representative.

Enjay Chemical company, New York, N. Y., has named Stephen L. Wythe as Company representative.

Denco Petroleum company of Cleveland, Ohio, has named E. E. Busse as both Company and Technical representative.

Battelle Memorial Institute of Columbus, Ohio, has named Richard L. Jentgen to the positions of both Company and Technical representative to NLGI. Mr. Jentgen is principal chemist of lubricants research.

FDA Delay on Labeling Act

On July 19, 1961, the Food and Drug Administration announced suspension of penalty provisions of the Federal Hazardous Substances Labeling Act until February 1, 1962, for all hazardous substances other than highly toxic, extremely flammable and flammable.

(On January 31, 1961, FDA extended the date of enforcement until August 1, 1961. The above represents a further extension.)

The Act became enforceable February 1, 1961, but authorized the Department of Health, Education, and Welfare to extend the effective date for not more than 18 months.

On April 29, 1961, proposed regulations were published in the Federal Register and comments were requested thereon. Numerous comments were received from associations, firms, and individuals during the 60 days specified for submitting comments. In addition, an open meeting was held in Washington on July 13 and 14, 1961, at which time oral statements were received.

F. E. Rosenstiehl of Texaco, a past president of NLGI and a member of the Institute's board of directors, and T. W. H. Miller, general manager of NLGI, were in attendance, where a statement was read and submitted to FDA officials, pointing out the hardships that literal interpretation of the Act would work on the lubricating grease and fluid gear lubricants industry. (A copy of the NLGI statement was mailed to members on July 17.)

Deputy Commissioner of Food and Drugs John L. Harvey said that ". . . time will be required to consider all the comments received and to issue final regulations."

Any further developments on the situation will be brought to the immediate attention of the membership.

The Federal Caustic Poison Act remains in full force and effect during the period of this extension for any article affected thereby.

SERVICE AIDS

Send Orders to: National Lubricating Grease Institute, 4638 Nichols Pkwy., Kansas City 12, Mo.

FOIL SEALS OF NLGI'S NEW PROMOTION CHARACTER—Aluminum foil stickers, measuring one by one-and-a-half inches, show NLGI's little man in full color . . . add a distinctive touch to correspondence, advertising, etc. Box of one thousand, $7.50, postage paid.

NLGI MOVIE — "Grease, the Magic Film," a 16-mm sound movie in color running about 25 minutes, now released. First print $300, second and subsequent orders $200 each (non-members add $100 to each price bracket).

REPRINTS — From the NLGI SPOKESMAN are available at low cost. Page forms are left standing for three months, company imprint or advertising arranged.

Terrific New DOOR OPENER

TRUCKER'S FIFTH WHEEL LUBRICANT

. . . in a convenient cartridge that Needs No Gun

Open the door to additional sales with the most sensational new packaging development on the grease market . . . the instant-applicator cartridge filled with Fifth Wheel Lubricant. Now . . . the trucker can carry a fresh lubricant supply with him, insuring uniformity of product and correct lubrication between maintenance intervals. It's the cleanest, fastest, most convenient method of lubricating truck fifth wheels ever introduced. A simple turn of the plastic ring releases the lubricant in perfect flow . . . a reverse turn stops the flow.

- **FAST AND EASY TO USE**
- **NO MESS — NO WASTE**
- **NO PRODUCT CONTAMINATION**
- **NEW CONVENIENCE FOR TRUCKERS**
- **ON THE SPOT LUBRICATION WHEN IT'S NEEDED**

Available Under Stock Label or Private Brand in Minimum Quantities

Trucker's Fifth Wheel Lubricant is a premium product fortified with Moly, graphite, and extreme pressure additives. It provides free turning and complete lubrication protection under all weather conditions. Fifth Wheel Lubricant prevents galling, seizing, gouging, and noisy metal to metal contact. It protects against rust and corrosion. Stores indefinitely . . . even after being opened.

Be the first in your marketing area to offer Trucker's Fifth Wheel Lubricant in the self-applicators.

WRITE, WIRE OR PHONE FOR COMPLETE INFORMATION

Battenfeld

BATTENFELD GREASE & OIL CORP., INC.
32nd and Roanoke Road, Kansas City, Missouri

Future Meetings

SEPTEMBER, 1961

11 NLGI Board of Directors meeting, Roosevelt Hotel, New York City.

13 API Division of Marketing, Lubrication Committee Meeting, Traymore Hotel, Atlantic City, N. J.

13-15 National Petroleum Association, Annual Meeting, Traymore Hotel, Atlantic City.

24-26 Independent Oil Compounders Association, Annual Meeting, Villa Moderne Motor Hotel, Highland Park, Ill.

24-28 ASTM Committee D-2 meeting, Statler Hotel, Detroit.

OCTOBER, 1961

17-19 ASME-ASLE Lubrication Conference, Hotel Morrison, Chicago.

18-20 Packaging Institute, 23rd Annual National Packaging Forum, Biltmore Hotel, New York City.

OCT. 29 - NOV. 1, 1961 NLGI Annual Meeting, Rice Hotel, Houston, Tex.

NOVEMBER, 1961

6-7 Petroleum Packaging Committee, Packaging Institute, Fort Shelby Hotel, Detroit, Mich.

9-10 SAE National Fuels and Lubricants Meeting, Shamrock-Hilton Hotel, Houston, Texas.

13-15 American Petroleum Institute Annual Meeting, Conrad Hilton Hotel, Chicago.

26-Dec. 1 ASME Winter Annual Meeting, Statler Hilton Hotel, New York.

JANUARY, 1962

8-12 Society of Automotive Engineers, Annual Meeting, Cobo Hall, Detroit, Mich.

FEBRUARY, 1962

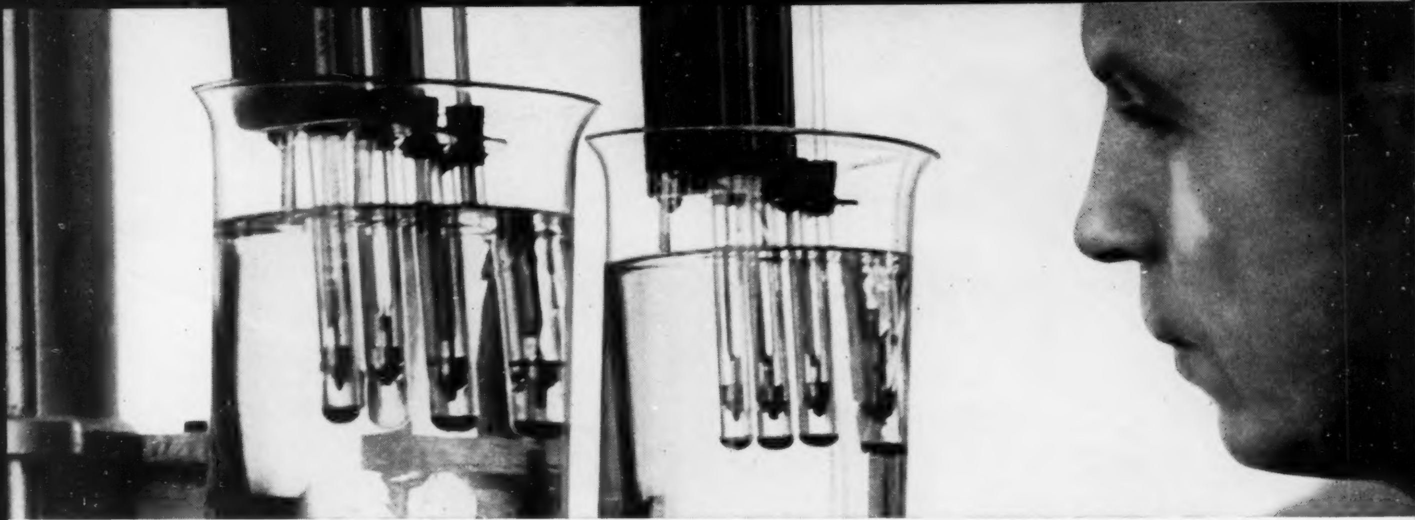
21 NLGI Board of Directors meeting, Sheraton-Cadillac Hotel, Detroit, Mich.

MAY, 1962

22-25 API Division of Marketing, Midyear Meeting, Queen Elizabeth Hotel, Montreal, Canada.

OCTOBER, 1962

21-24 NLGI Annual Meeting, Edgewater Beach Hotel, Chicago.



Dropping point test shows how greases react to heat. Beaker fluid has been heated to 390°F. All greases tested except Darina (second tube from let) have passed from solid to liquid state.

BULLETIN:

Shell reveals the remarkable new component in Darina Grease that helps it save up to 35% on grease and labor costs

Darina® Grease is made with Microgel*, the new thickening agent developed by Shell Research.

Darina lubricates effectively at temperatures 100° hotter than most conventional soap base greases can withstand.

Read how this new multi-purpose industrial grease can help solve your lubricating problems and even save you up to 35% on grease and labor costs.

THERE IS no soap in Darina Grease. No soap to melt away—wash away—or dissolve away.

Instead of soap, Darina uses Microgel—a grease component developed by Shell Research.

What Microgel does

Because of Microgel, Darina has no melting point. It won't run out of gears or bearings.

Compared with most conventional soap-base greases, Darina provides significantly greater protection under adverse service conditions.

Mix water into Darina and the grease does not soften. It shrugs off water—won't emulsify.

Resists heat

Darina will withstand operating temperatures 100° hotter than most conventional multi-purpose greases. It cuts leakage and reduces the need for special high-temperature greases.

Also, Darina resists slumping, thus forming a more effective seal against foreign matter.

Saves money

Shell Darina can reduce maintenance expenses while it protects your machinery. Savings of up to 35% on grease and labor are quite possible.

In some cases lubrication intervals have been extended to double what they were before. Less grease is consumed and less time consumed applying it.

For details, see your Shell Representative. Or write: Shell Oil Company, 50 West 50th Street, New York 20, New York.

*Registered Trademark

SHELL

A BULLETIN FROM SHELL
—where 1,997 scientists are helping to provide better products for industry

Come to Houston . . .

New Oil Capital of the World!

NLGI 29th ANNUAL MEETING

Rice Hotel, October 29-November 1, 1961

A perfect convention combination . . . the hospitality of the fabulous Texans and a lubricants program with the latest in technical, marketing and manufacturing information . . . all at the 29th Annual Meeting of the National Lubricating Grease Institute, the Rice Hotel, Houston, Texas.

Beginning with the "Mr. and Mrs. Early Bird Reception" Sunday evening, October 29, right on through to the Technical Committee meeting on Wednesday morning, November 1, there is an excellent selection of meetings, symposiums, sessions formal and informal, social events, and a welcome to Houston as pleasant as the weather.

There'll be activities for the ladies, too, with an air-conditioned boat ride down to the famous San Jacinto Inn as just one of the high spots.

The meetings will feature reports on rapeseed oil lubricating greases, a new look at colloidal graphite, the nature of acetate complexes in greases, and pilot scale manufacture of lubricating greases.

Marketing men will be particularly interested in the presentation on how to gain empathy between researcher and marketer, a topical look at the past and the future, and one of the most thorough farm lubrication papers ever given.

The Federal Hazardous Substances Labeling Act will come under scrutiny again, as one of the foremost experts on the subject explains how this legislation affects the entire industry.

Manufacturing will cover modern grease plant layout, machinery for the the new plant, modern warehousing methods and product distribution, and new aspects in grease milling.

The ASTM-NLGI symposium on oil viscosity, consisting of five papers, will be another attraction . . . this will be a concurrent session, running with the marketing meeting.

The business of the Institute will be conducted, too. There will be two board of directors meetings, the annual business meeting, the steering committee meeting and group sessions by all of the subcommittees. Finally, the Technical Committee meeting will round up a productive program.

Besides the special social events for the ladies, there will be the Early Bird reception, while Tuesday even-

ing, October 31, will be given over to the traditional Social Hour and Annual Banquet. Following the NLGI Annual Meeting your attention is drawn to the Galveston area nearby, Old Mexico is just a few hours away, and the API-Louisiana State University farm lubrication conference (November 2 and 3). It all adds up to a most productive time, so head for Houston and the Rice Hotel.

Schedule of NLGI Annual Meeting Papers

General Sessions

PRESIDENT'S ADDRESS, *F. R. Hart*, Standard Oil Co. (California), San Francisco.

KEYNOTE ADDRESS: FLUID GEAR LUBRICANTS—THEIR FUTURE, *Henry Korp, Vice-President*, Southwest Research Institute, San Antonio, Tex.

RAPESEED OIL LUBRICATING GREASES, *I. S. Evans, J. P. Pardo, M. A. McNichol*, Saskatchewan Research Council, Sta-Vis Oil, Federated Co-operatives, Ltd., Saskatoon, Minneapolis and Regina.

DEVELOPMENTS IN COLLOIDAL GRAPHITE, *E. L. Youse*, Joseph Dixon Crucible Co., Jersey City, N. J.

NATURE OF ACETATE COMPLEXES IN GREASES, *J. Panzer*, Esso Research and Engineering, Linden, N. J.

PILOT SCALE MANUFACTURE OF LUBRICATING GREASE, *E. J. Blake*, British Petroleum, Middlesex, England.

Market Sessions

CREATIVITY IS WHERE YOU FIND IT, *H. L. Hemmingway*, Pure Oil, Crystal Lake, Ill.

A THREE-DECADE REVOLUTION, *C. J. Boner*, Battenfeld Grease & Oil, Kansas City.

FEDERAL HAZARDOUS SUBSTANCES LABELING ACT, *A. E. Dooley*, Texaco, New York.

MARKET RESEARCH: A KEY TO PROFITS IN FARM LUBRICANTS, *R. E. Price*, Consumers Cooperative, Kansas City.

Manufacturing Sessions

MODERN GREASE PLANT LAYOUT, *C. W. Nofsinger*, C. W. Nofsinger Co., Kansas City.

MACHINERY FOR THE NEW PLANT, *G. J. Barrett, Jr.*, Barrett Manufacturing, Houston.

NEW ASPECTS OF GREASE MILLING AND MILLING EQUIPMENT, *L. E. Putnam*, Chemicolloid Laboratories, Garden City Park, N. Y.

ASTM-NLGI Symposium on Oil Viscosity

MASTER CURVES FOR GREASE FLOW, *L. C. Brunstrum, A. C. Borg, A. W. Sisko*, American Oil, Whiting, Ind.

THE ROLE OF BASE OIL VISCOSITIES IN PERFORMANCE OF ELECTRIC MOTOR GREASES, *K. H. Warren*, U. S. Naval Engineering Experiment Station, Annapolis, Md.

PROBLEMS ENCOUNTERED IN CENTRALIZED LUBRICATION SYSTEMS AT LOW RATE OF SHEAR, *A. C. Allen, C. F. Raisch*, Stewart Warner, Chicago.

THE EXPERIENCE OF THE ORDNANCE CORPS WITH GREASES MADE FROM LOW VISCOSITY OILS, *S. F. Calhoun*, Rock Island Arsenal, Rock Island, Ill.

THE EFFECT OF VARIATIONS IN THE VISCOSITY AND TYPE OF THE MINERAL OIL COMPONENT ON OIL SEPARATION FROM GREASES OF A LITHIUM-CALCIUM SOAP, *J. L. Zakin, G. W. Murray, Jr.*, Socony Mobil Oil, Brooklyn.

LUBRICATION AND DESIGN OF SLEEVE BEARINGS, *C. L. Pope*, Eastman Kodak Co., Rochester, N. Y.

AN APPROACH FOR RESEARCH IN GREASE LUBRICATED CAST BRONZE BEARINGS, *C. N. Paden, Jr.*, Moccasin Bushing Co., Chattanooga, Tenn.

OPERATING TECHNIQUES IN SOAP MAKING, *W. A. Graham*, Stratford Engineering Corp., Kansas City.

OTHER ANNUAL MEETING HIGHLIGHTS include subcommittee meetings, steering committee action, the annual business meeting, and the NLGI Technical Committee meeting.

NLGI 29TH ANNUAL MEETING, RICE HOTEL, HOUSTON, TEXAS, OCTOBER 29—NOVEMBER 1, 1961. REGISTRATION SEVEN DOLLARS (MEMBERS) AND TWELVE DOLLARS (NON-MEMBERS), BANQUET TEN DOLLARS AND SOCIAL HOUR THREE DOLLARS. FOR FURTHER INFORMATION PLEASE CONTACT: NATIONAL LUBRICATING GREASE INSTITUTE, 4638 J. C. NICHOLS PARKWAY, KANSAS CITY 12, MISSOURI. (PHONE) VALENTINE 1-6771. T. W. H. MILLER, GEN. MGR.

Revised NLGI GLOSSARY

About the Author

A. S. Orr, chairman of the Subcommittee on Manual of Test Methods & Definitions of Terms Peculiar to the Lubricating Grease Industry, obtained a BS degree in chemical engineering from Texas A&M college in 1924. Immediately after graduation, he joined Gulf Oil corporation in the Port Arthur, Tex., laboratory. In 1930 he was transferred to Gulf's refinery technology laboratory in Philadelphia, Pa., and became director of that laboratory in 1950. In 1951 he was transferred to Gulf's main office in Pittsburgh, Pa., and is currently working in the coordinator's group of the manufacturing department handling matters pertaining to product development and product quality. Much of this work has to do with grease development and quality. Mr. Orr is a member of the American Chemical Society, SAE, ASTM and other technical societies. He has contributed to the Spokesman *before.*

New Additions to Terms and Definitions Made by Subcommittee

By: A.S. Orr

Gulf Oil Corporation

THE NLGI Technical Committee, through one of its subcommittees, has been working for over five years in developing definitions of terms relating to the lubricating grease industry. All definitions approved previously have been published in earlier issues of the NLGI Spokesman, and were collected and published as a Glossary by NLGI in 1960.

Listed below are additional definitions on which substantially unanimous approval has been obtained during the year 1960. Several definitions proposed last year were not acceptable to all members of the Technical Committee and these are being studied further by the Subcommittee. Also, a group of definitions of some terms not previously considered will be proposed for consideration at the next annual meeting of NLGI.

Several of the following definitions relate to properties of lubricating greases which are evaluated entirely by visual observation. Such observations are made frequently by both users and producers of greases, and often influence acceptance of a grease as much as other properties requiring elaborate test equipment to evaluate. Thus, it is particularly important that all of us mean the same thing when we speak of such properties as "appearance" or "texture," and that we use the same terms in describing various aspects of these properties.

During the past year the Subcommittee on Definitions of Terms Related to Lubricating Greases was made up of:

A. S. Orr, Chairman, Gulf Oil Corporation
C. E. Hulme, Vice-Chairman, Kendall Refining Company
J. W. Blattenberger, Cities Service Res. & Dev. Co., Inc.
C. J. Boner, Battenfeld Grease & Oil Company
M. L. Carter, Southwest Grease & Oil Company, Inc.
J. J. Dickason, Jesco Lubricants Company
W. J. Eubank, Cato Oil & Grease Company
H. U. Fisher, California-Texas Oil Company
E. M. Higgins, Master Lubricants Company
J. D. Smith, International Lubricant Corporation

Recently Mr. M. L. Langworthy, Caltex Service Company, has replaced Mr. H. U. Fisher on the Subcommittee.

In addition to the members of the Subcommittee who are primarily responsible for the definitions listed below, many other members of the NLGI Technical Committee have submitted constructive criticism and helpful suggestions on proposed definitions. These were invaluable in arriving at acceptable definitions and are gratefully acknowledged.

Definitions of Terms Relating to the Lubricating Grease Industry

ADDITIVE—any material added to a lubricating grease or lubricating oil to improve its suitability for service. It may improve a property already possessed by the lubricant or give it properties not naturally possessed. (Additives are the "other ingredients imparting special properties" referred to in the definition of lubricating grease. Typical examples are anti-oxidants and "EP" or anti-weld additives.)

AGE HARDENING—increase in consistency (hardening) of a lubricating grease with storage time.

APPARENT VISCOSITY—the ratio of shear stress to rate of shear of a non-Newtonian fluid, calculated from Poiseuille's equation and measured in poises. (Changes with changing rates of shear and temperature, and must therefore be reported as the value at a given shear rate and temperature.)

APPEARANCE—Those characteristics of a lubricating grease which are observable by visual inspection only. This general term includes various characteristics described under BULK APPEARANCE, THIN FILM APPEARANCE, BLOOM, COLOR, and LUSTER.

BULK APPEARANCE—What a lubricating grease looks like when the undisturbed surface is viewed in an opaque container. BULK APPEARANCE should be described in the following terms:

SMOOTH—A surface relatively free of irregularities.

ROUGH—A surface composed of many small irregularities.

CRACKED—Showing surface cracks of appreciable magnitude. In describing such a lubricating grease, the number and size of the cracks should be included in the description.

BLEEDING—Showing free oil on the surface of the lubricating grease (or in the cracks of a cracked grease).

BLOOM—The surface color (usually blue or green) of a lubricating oil or grease when viewed by reflected daylight at an angle of about 45 degrees from the surface. BLOOM is associated with the absorption of ultra violet light in the oil and may not be visible if the sample is viewed by artificial light.

COLOR—(Of Lubricating Grease) The shade and intensity shown when lubricating grease is viewed under conditions such as to eliminate BLOOM.

Such conditions may be obtained by viewing the lubricating grease in an opaque container such as a metal package, by reflected light, from a position approximately perpendicular to the surface; or by viewing it with transmitted light only, by placing the sample on a transparent plate. When colors of lubricating grease are referred to, the method by which colors are determined should be clearly indicated.

Colors of lubricating greases are best described in terms of the predominant hue such as amber, brown (or perhaps green, red, or blue for dyed greases) with a qualifying adjective describing intensity in terms of light, medium, or dark.

COLOR (of lubricating grease)—See under APPEARANCE.
(of lubricating oil)—That shade shown when viewed under transmitted light only. Usually lubricating oil colors are obtained by viewing the oil under specified conditions in test equipment. Several such methods are available, the most widely used being that of ASTM Method D 1500 which describes the colors in terms of numbers.

BLEEDING—the separation of liquid lubricant from a lubricating grease for any cause. (While excessive bleeding is harmful, a small amount of free oil can aid in lubrication.)

CAVITATION—in a lubricating grease dispensing system, failure for any reason of the material to flow to the suction of the system pump. (See also FEEDABILITY.)

CHANNELING—1. a term used in connection with lubricating greases to describe the (usually desirable) tendency to form a channel by working down of lubricating grease in a bearing, leaving shoulders of unworked grease which serve as seal and reservoir.

2. a term used in connection with liquid lubricants and flow type lubricating greases to describe the tendency at low temperatures, for these materials to form a plastic structure sufficiently strong to resist flow under gravitational forces only. (Similar to, but not identical with the pour point of liquid lubricants, it is measured by empirical tests such as Federal Method 3456-T.)

COLD SETTS—see SETT GREASE.

COMPLEX SOAP—a soap wherein the soap crystal or fiber is formed by co-crystallization of two compounds:

1. the normal soap (such as metallic stearate or oleate).

2. the complexing agent. (Examples of complexing agents are the metallic salts of short chain organic acids such as acetic or lactic, or the inorganic salts such as the carbonate or chlorides. Still another complexing agent is water, in which case the soap may also be referred to as a HYDRATED SOAP.)

CONSISTENCY (HARDNESS)—the degree to which a plastic material such as lubricating grease resists deformation under the application of force. (It is therefore a characteristic of plasticity, as viscosity is a characteristic of fluidity. Consistency is usually indicated by either apparent viscosity or ASTM penetration.)

CORROSION—the gradual destruction and/or pitting of a metal surface due to chemical attack. This chemical attack may be, but is not necessarily, due to the formation of acidic materials in the lubricant. See also FRETTING.

DISPENSABILITY—the property of a grease which governs the ease with which it may be transformed from its container to its point of application. Most used in discussion of dispensing in grease systems, where it includes both the properties of pumpability and feedability.

DROPPING POINT—the temperature at which a drop of material falls from the orifice of the test apparatus under the conditions of ASTM test D566-42. (This test should be considered as having very limited bearing upon service performance. It is not the melting point of the grease, this being a term of little or no significance, when applied to plastic materials such as lubricating grease. These materials are characterized by a *melting* range during which the material becomes steadily softer. Only through the establishment of an arbitrary and fully controlled test procedure such as the ASTM Dropping Point, can any single temperature be established as a characteristic of the material. A similar test, differing only in details, the IP "Drop Point" usually gives a lower value.)

FALSE BRINELLING—See FRETTING.

FEEDABILITY—the ability of a lubricating grease to flow to the suction of a dispensing pump at a rate at least equal to pump delivery capacity. (Some lubricating greases do not feed satisfactorily and cause cavitation at the inlet to a dispensing pump. In such cases, feedability can often be made satisfactory by the use of follower plates.)

FIBER—in lubricating grease, the form in which soap thickeners occur, the soaps crystallizing in threads which are of the order of 20 or more times as long as they are thick. (Most soap fibers are microscopic in size, so that the grease appears smooth to the eye. The greases having FIBROUS appearance are those where the fiber bundles are large enough to be seen by the naked eye. The most common fibrous lubricating grease is sodium base, although not all sodium base greases are fibrous.) See also APPEARANCE and TEXTURE.

FIBRIL—an extremely small fiber, usually barely visible even at maximum magnification of the electron microscope. Fibrils may collect in bundles to form the larger fibers.

FOLLOWER PLATE—a plate fitted to the top surface of lubricating grease in a container and so designed that as lubricating grease is dispensed, atmospheric pressure assists gravitational forces in delivering grease to the inlet of the dispensing system.

FRETTING—the removal of very finely divided particles from bearing surfaces due to inherent adhesive forces which are assisted by vibrating motion. The finely divided wear particles are then usually oxidized to a corrosion product which has led it to be called FRETTING CORROSION, although corrosion does not enter into the basic mechanism. It has also, in the past, been called FALSE BRINELLING and FRICTION OXIDATION when it occurred in rolling contact bearings.

FRETTING CORROSION—See FRETTING.

FRICTION OXIDATION—See FRETTING.

HOMOGENIZATION—the process of subjecting a lubricating grease to intimate mixing and intensive shearing action, the end result of which is to obtain a more uniform and higher degree of dispersion.

INCOMPATIBILITY—two lubricating greases show incompatibility when a mixture of the products shows physical properties or service performance which are markedly inferior to those of either of the greases before mixing. Performance or properties inferior to one of the products and superior to the other may be due to simple mixing and would not be considered as evidence of incompatibility.

INORGANIC THICKENER—See NON-SOAP THICKENER.

INSOLUBLES—Components of a lubricating grease which are insoluble in the prescribed reagents in an analytical procedure such as ASTM D 128. The analytical procedure used should be indicated when insolubles are specified. Additional identifying analytical tests are required to determine the nature and composition of insolubles which may consist of fillers, additives, certain types of thickeners, or inadvertent impurities.

LUBRICATING GREASE STRUCTURE—the physical arrangement of the component particles of a lubricating grease thickener, additive and liquid lubricant. It is the nature and stability of this arrangement which determine the appearance, texture and physical properties of lubricating grease.

LUSTER—The intensity of light reflected by lubricating grease; its sheen or brilliance. LUSTER should be described as follows:

BRIGHT—Reflects light with a relatively strong intensity.

DULL—Reflects light with a relatively weak intensity. Some greases with a high water content may have a dull luster. Certain thickeners and fillers give a grease a characteristic dull luster.

MECHANICAL STABILITY—See SHEAR STABILITY.

NEWTONIAN BEHAVIOR—the property of simple liquids by which the rate of shear is proportional to the shearing stress. This constant proportion is the viscosity of the liquid.

NLGI NUMBER—a numerical scale for classifying the consistency range of lubricating greases, and based on the ASTM penetration number. NLGI grades are in order of increasing consistency (hardness) as follows:

NLGI NUMBER	ASTM Worked Penetration
0	355-385
1	310-340
2	265-295
3	220-250
4	175-205
5	130-160
6	85-115

(Greases both softer and harder than this consistency range are well known in the industry. Such greases do not bear an NLGI number.)

NON-NEWTONIAN BEHAVIOR—the property possessed by some fluids and many plastic solids including lubricating grease, of having a variable relationship between shear stress and rate of shear. (Non-Newtonian materials, therefore, *do not possess a viscosity* as defined by Newton, but rather an apparent viscosity, the quantitative value of which may vary widely with varying shear rate. Conventional types of viscometers with uncontrolled shear rates will not satisfactorily measure non-Newtonian materials.)

NON-SOAP THICKENER—any of several specially treated or synthetic materials, excepting the metallic soaps of long chain fatty acids, which can be either thermally or mechanically dispersed in liquid lubricants to form lubricating grease. Also called SYNTHETIC THICKENER. Certain types are called INORGANIC THICKENERS.

PENETRATION—an arbitrary measure of consistency (hardness), based on ASTM Method D217-52T (and similar methods standardized by other organizations). (All penetration measurements are in an inverse scale of consistency—that is, the softer the consistency, the higher the penetration number.)

The ASTM definitions are given as follows:

PENETRATION of lubricating grease is the depth, in tenths of a millimeter, that a standard cone penetrates the sample in a standard cup under prescribed conditions of weight, time and temperature. The penetration depends on whether or not the consistency has been altered by handling or agitation.

WORKED PENETRATION is the penetration of a sample of lubricating grease immediately after it has been brought to 77 F and then subjected to 60 strokes in a standard grease worker.

UNWORKED PENETRATION is the penetration at 77 F of a sample of lubricating grease which has received only the minimum handling in transfer from sample can to test apparatus and which has not been subjected to the action of a grease worker.

BLOCK PENETRATION is the penetration at 77 F of a sample of lubricating grease that is sufficiently hard to hold its shape.

PLASTICITY—that property of apparently solid material which enables it to be permanently deformed under the application of force, without rupture. (Plastic flow differs from fluid flow in that the shearing stress must exceed a yield point before any flow occurs.)

PUMPABILITY—the ability of a lubricating grease to flow under pressure through the line, nozzle and fitting of a grease dispensing system. It is best indicated by the apparent viscosity at moderate rate of shear. (See NLGI Tentative Method for Matching Lubricating Grease Flow Properties with Lubricating Grease Dispensing Pump Delivery Behavior at Low Temperatures.)

RHEOPECTIC GREASE—a lubricating grease which has the property of increasing in consistency, (hardening appreciably) upon being subjected to shear.

SETT GREASE—a lubricating grease made by mixing a mineral oil slurry of an alkali and a mineral oil solution of a fatty acid, saponification usually being completed after the mixture is poured into the shipping container. As the temperature of the mixture is generally less than 140 F, these products are sometimes known as COLD SETTS. Axle grease is usually a SETT GREASE.

SHEARING—slipping or sliding of one part of a substance relative to an adjacent part. In a solid, such action involves cutting or breaking of the crystal structure, but in a fluid or plastic, shearing does not necessarily destroy the continuous nature of the substance.

SHEAR RATE—the rate of slip within a substance engaging in flow. The average or *mean* shear rate in a pipe or tube is the average velocity divided by the radius of the tube. It, therefore, has the dimensions of the reciprocal of time and is usually expressed in the unit of reciprocal seconds (sec^{-1}). The mean shear rate is reported in the determination of apparent viscosity in ASTM Method D1092-55.

SHEAR STABILITY—the ability of a lubricating grease to resist changes in consistency (hardness) during mechanical working. Working may be in any of several types of laboratory machines or may be in actual service. This may also be called MECHANICAL STABILITY.

SHEAR STRESS—the force required to cause shearing in a substance. In fluids, the relation of the shear stress to the shear rate is the viscosity of the substance.

SOLID LUBRICANT—any class of lubricants wherein the reduction of friction and wear during sliding is caused by making the shearing take place within the crystal structure of a material with low shear strength in one particular plane. Examples include graphite, molybdenum disulfide and certain soaps. Lubricating grease is *not* a solid lubricant, but may contain solid lubricants as additives.

SYNERESIS—loss of liquid lubricant from a lubricating grease due to shrinkage or rearrangement of the structure. The shrinkage may be due to either physical or chemical changes in the thickener. Syneresis is a form of BLEEDING.

SYNTHETIC THICKENER—See NON-SOAP THICKENER.

TEXTURE—That property of lubricating grease which is observed when a small separate portion of it is pressed together and then slowly drawn apart. TEXTURE should be described in the following terms:

> BRITTLE—Has a tendency to rupture or crumble when compressed.
>
> BUTTERY—Separates in short peaks with no visible fibers.
>
> LONG FIBER—Shows tendency to stretch or string out into a single bundle of fibers.
>
> SHORT FIBER—Shows short break-off with evidence of fibers.
>
> RESILIENT—Capable of withstanding moderate compression without permanent deformation or rupture.
>
> STRINGY—Shows tendency to stretch or string out into long fine threads, but with no visible evidence of fiber structure.

Other terms such as SMOOTH, ROUGH, GRAINY, etc., are defined under APPEARANCE.

THICKENING AGENT—the solid particles which are relatively uniformly dispersed to form the structure of lubricating grease in which the liquid is held by surface tension and other physical forces. (The solid particles may be fibers, as is the case with various metallic soaps, or plates of spheres, as is the case with some of the non-soap thickeners. The only general requirements are that the particles should be extremely small and that they be capable of uniform dispersion in the liquid lubricants.)

THIXOTROPY—in lubricating grease, that property which is manifested by a decrease in cosistency, or softening, as a result of shearing, followed by an increase in consistency, or hardening, beginning after shearing is stopped. (Thixotropic age hardening is a relatively prolonged process proportional to aging time, and is seldom if ever, complete, whereas the apparent viscosity increase which occurs in non-Newtonian systems with decreasing shear rate is instantaneous and fully reversible. Lubricating grease is *both* thixotropic and non-Newtonian.)

WATER RESISTANCE—The ability of a lubricating grease to withstand the addition of water to the lubricant system without adverse effects. WATER RESISTANCE is generally considered to be made up of three components as listed below:

> WASHOUT RESISTANCE—The ability of a lubricating grease to resist being removed from a bearing when acted on by a stream of water. (Generally measured by ASTM Method D 1264.)
>
> WATER ABSORPTION CHARACTERISTICS—The characteristics of a lubricating grease when water is added to the lubricant system. WATER ABSORPTION CHARACTERISTICS may be measured by any of several suitable tests in which the lubricating grease may react in any of three ways, described as follows:
>
> > WATER SOLUBLE—The lubricating grease absorbs the water, and then de-gels to a semifluid consistency.
> >
> > WATER ABSORBENT—The lubricating grease absorbs relatively large quantities of water with small or no change in consistency, and without leaving free water as a separate phase.
> >
> > WATER RESISTANT—The lubricating grease does not absorb more than small amounts of water, does not change appreciably in consistency, and leaves the added water as a second phase in the system.
>
> WATER CORROSION RESISTANCE—The ability of a lubricating grease to prevent corrosion of surfaces when water is present in the lubricating system. May be measured either statically by any of a number of standard tests, or dynamically by actual operation of bearings with water added to the lubricant reservoir.

Lubricating greases for various types of service may need any of the several types of WATER RESISTANCE characteristics described above, so that they are not measures of quality except for specific situations where particular properties are required.

WORKING—subjecting lubricating grease to any form of agitation or shearing action.

YIELD (OF LUBRICATING GREASE)—the amount of grease of a given consistency which may be made with a definite amount of thickening agent. As the yield increases, % thickener decreases.

YIELD POINT (OR YIELD VALUE)—the minimum force required to produce flow of a plastic material. It is estimated by the intercept on the shear stress axis of the shear stress-shear rate curve, by extrapolation of the straight portion of the curve. ■

Electron Microscope Examination Of Thin Sections of Lubricating Grease Thickeners

By: J. W. Wilson, Jr.
Socony Mobil Oil Company, Inc.

Presented at the NLGI 28th annual meeting in Chicago, October, 1960

Introduction

For some thirty years it has been recognized that the structure and arrangement of the thickener particles in lubricating greases made a major contribution to their properties. Determining this structure in detail has been a continuing objective of lubricating grease research during this period. First attempts were based on conventional microscopy using both bright and dark field illumination and polarized light. While some gross structures were observed, it was soon apparent that the size of the thickener particles was usually below the limit of resolution of the conventional microscope. The advent of the electron microscope in the late 1930's and early 1940's removed this restriction and in 1947 Farrington and Birdsall[5] and in 1951 Farrington[6] discussed the application of this instrument to lubricating grease thickener structure. Since then the electron microscope has played a prominent role in research in this field. It has been supplemented by phase contrast microscopy, differential thermal analysis, X-ray and electron diffraction, and infrared spectroscopy. These methods are complementary and have made contributions toward the solution of thickener structure problems.

The electron microscope has given a great deal of information on the size and shape of the thickener particles. These geometric attributes have been correlated with a number of performance properties of the greases. However, performance differences have also been noted between greases showing very similar thickener structures. These differences have been attributed to differences in the space arrangement of the thickener particles. The conventional method of preparing the grease for electron microscope examination, namely air-drying of a petroleum ether suspension, destroys the space arrangement of the thickener particles. The Volds *et al.*[10, 12] made a start on this problem by cutting thin sections from frozen cetane-thickener gels. McClellan and Cortes[7] developed an aerogel technique for sample preparation which gave additional information on thickener structure. The Volds' specimens approached the maximum thickness which can be examined in the electron microscope because of the

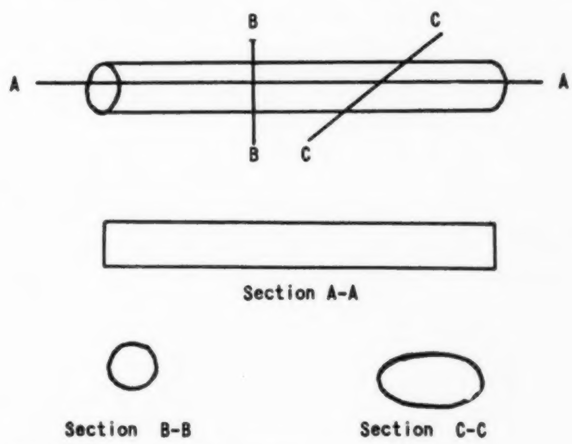


FIGURE 1—Simplest theoretical case—a cylinder.

limitations of the microtomes then available. Improved microtomes have since been built and ultrathin sectioning has become commonplace in biological and medical investigations. A modification of the method of Borysko[3,4] has been used in the present investigation. It has proved adaptable and convenient for the precision microtomy of lubricating greases. Procedures and apparatus used in this work are given in the Appendix.

Results and Discussion

1. *Appearance of Sections*

Viewing a section cut with a microtome allows us to see a particular plane through a three-dimensional structure. However, the development of the structures seen in the plane from the actual structures present in the bulk material is often difficult to visualize. This simply is not our customary way of viewing objects. This problem becomes acute when we discuss lubricating grease thickeners or other microscopic particles. In this case, we cannot even see the bulk material with the naked eye. However, we do have a good idea of the size and shape of individual particles of the thickener from previous electron microscope investigations. These have shown that many of these particles are in the form of fibers. Assuming for simplicity that we are dealing with uniform fibers, we can orient our thinking by means of diagrams to show what the appearance of sections might be.

The simplest form of a fiber, as shown in Figure 1, is a right cylinder. Three types of section can be cut from this cylinder. Cutting perpendicular to the plane of the paper along lines A-A, B-B, and C-C and then rotating the flat surfaces so cut through 90° around the cylinder axis into the plane of the paper, we would have the corresponding sections. The sections would have the same shape when viewed perpendicular to the plane of the cut.

The fibers in lubricating grease thickeners actually exist in more complicated forms. Figure 2 shows the appearance of sections cut from a number of hypothetical fiber arrangements. We are again assuming a microtome cut perpendicular to the plane of the paper followed by rotation through 90° so the section can be viewed in the plane of the paper. The first case shows a section cut from a bent fiber which has a round bump on its surface. Assuming that this fiber lies entirely along the plane of the paper, then the three structures visible in the section, two ovals from the fiber and a circle from the bump, would lie in a straight line, as shown. However, it is more probable that the fiber will not lie in a single plane. A section through such a fiber is shown in Case 2. Note that the bend at the left end of the fiber is not visible in the conventional view and the various structures seen in the section do not line up in a straight line. Case 3 shows one type of section that would result from cutting through a network of fibers. The sections which result

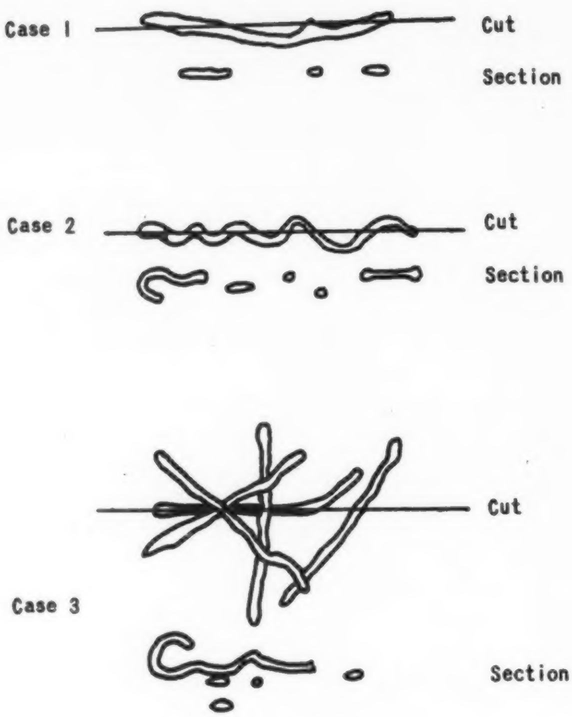


FIGURE 2—Some possible real cases.

from Case 2 and Case 3 have certain obvious similarities. In many actual cases it is impossible to reconstruct the actual situation without serial sections. Much information, however, can be obtained from single sections, providing their limitations are given proper consideration.

2. *Interpretation of Sections*

The above discussion indicates that the examination of a single section does not give an unequivocal determination of the structure from which the section was cut. The comments on the electron micrographs of sections which follow represent our thoughts after examining a large number of individual sections. They are, however, subject to review as further samples are examined and especially as techniques are developed for obtaining serial sections.

One other factor must also be taken into consideration. With the electron microscope, photographing the entire area of one grid at even relatively low magnifications requires an excessive number of pictures and shows a great deal of repetition. As a result, pictures are selectively taken of the area examined. There is then a tendency in a paper of this type to present only the most aesthetically gratifying and photographically correct prints, giving a selection of a selection. Thus, the viewing of a single electron micrograph can give a false impression of an over-all picture of a specific subject. Actually, this may or may not be the

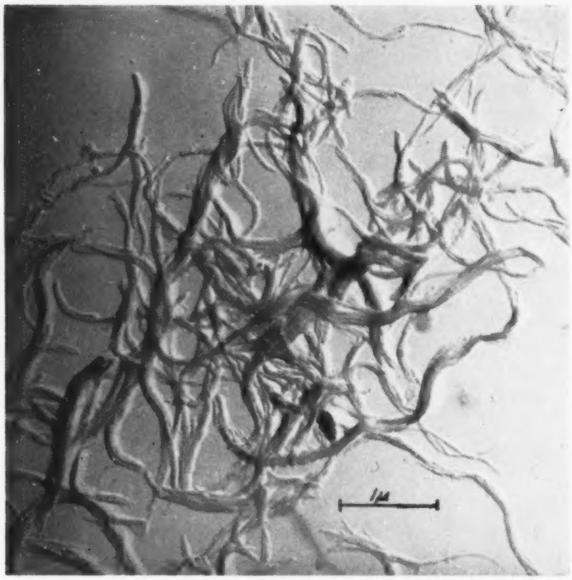


FIGURE 3A—Lithium-calcium grease. Conventional preparation from P.E. suspension, chromium shadowed at ca 20°.

case, dependent on the subject. The real over-all picture actually resides with the microscopist who, of necessity, must scan several grids of a single material in order to secure representative pictures.

3. *Lithium-Calcium Grease*

Figure 3 shows a conventional electron micrograph of the thickener in the lithium-calcium grease used in these studies. This specimen was prepared from a suspension of the lubricating grease in petroleum ether. This preparation results in a destruction of the space relationship of thickener particles present in the original grease. As a result, the relationships of the fibrous structure in Figure 3 are different from those present in the original grease. Except for certain special cases (e.g. twisted fibers) there is considerable evidence, however, that the size and shape of the individual particles are not changed to any serious extent by this process. Plates A, B, C are shown so that a general idea can be obtained of the size distribution of the fibers in the specimen.

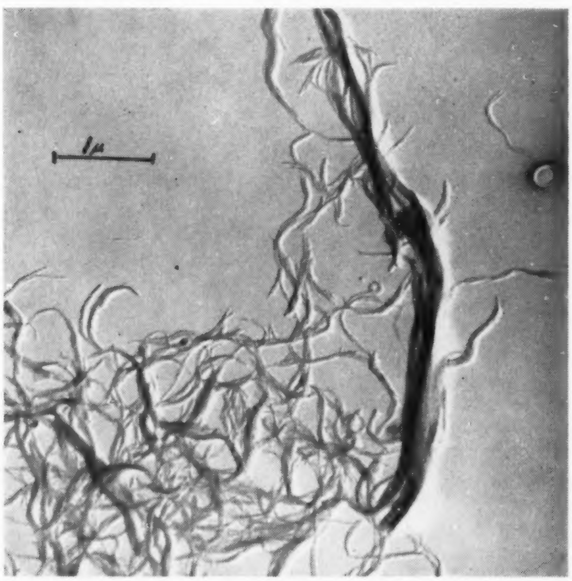


FIGURE 3B—Lithium-calcium grease. Conventional preparation from P.E. suspension, chromium shadowed at ca 20°.

Figure 4 shows a section cut along a fiber. The most striking features of this fiber are its particulate nature and its discontinuous edges. Also the variable density of the staining indicates a non-uniform cross section.

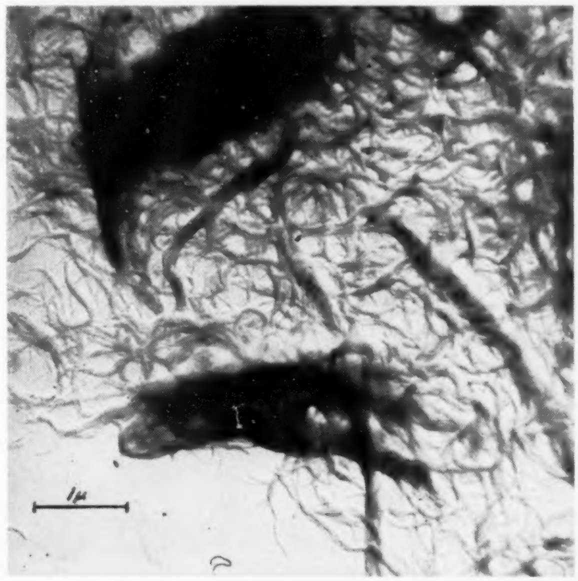


FIGURE 3C—Lithium-calcium grease. Conventional preparation from P.E. suspension, chromium shadowed at ca 20°.

The fiber appears to have been canted at a very small angle to the face of the cutting blade. It appears to be cut through almost parallel to the knife in the area of the loop or bend, but at the tail the fiber is disappearing either above or below the section.

Figure 5 shows another section cut from the methacrylate embedding of the lithium-calcium grease thickener. Area A shows a section through a fiber. Note especially the stained particles along the edges of this fiber. Similar particles can be seen along the edges of the fiber in Figure 4. Area B also contains what is probably another fiberlike organization as shown by increased density. The significance of this increased density can be seen in Figure 6. Area D shows a number of small particles between fibers. Area C is shown at a higher magnification in Figure 6.

Area A in Figure 6 shows a build-up of small particles between more densely stained larger particles. This is probably a section near the surface of a fiber. The B areas show concentrations of small particles and fibrils adjacent to larger particles or fibers. These small bits of material may play an important part in fiber formation, as will be discussed later. Area C shows a very small fiber similar to the one shown in Figure 4.

Figure 7 shows an area similar in over-all appearance to Figure 6. However, the coordination of areas into fiber systems is more apparent. The areas labeled A show sufficient structure to indicate a fiber cross section. In contrast, the other areas in this section are almost clear, i.e. devoid of any structure related to the grease. The areas labeled B show large numbers of small particles or fibrils associated with larger particles.

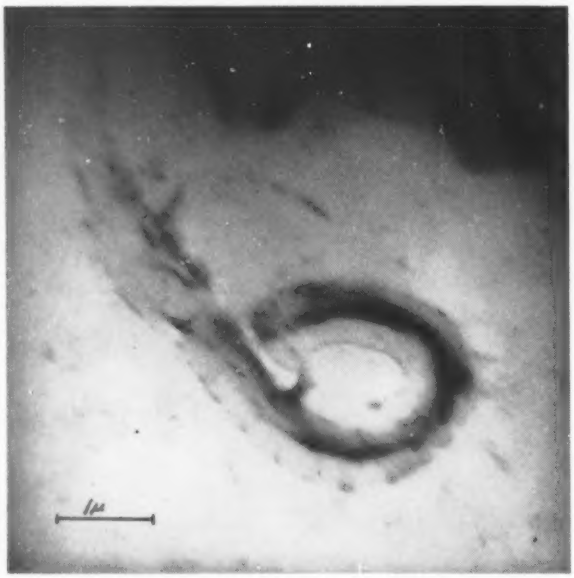


FIGURE 4—Lithium-calcium grease, methacrylate section.

Figure 8 shows considerable internal structure in fiber cross sections. One section in particular shows a repeated or periodic structure. This should be compared with Figure 9. Figure 8 also shows another section cut along a fiber.

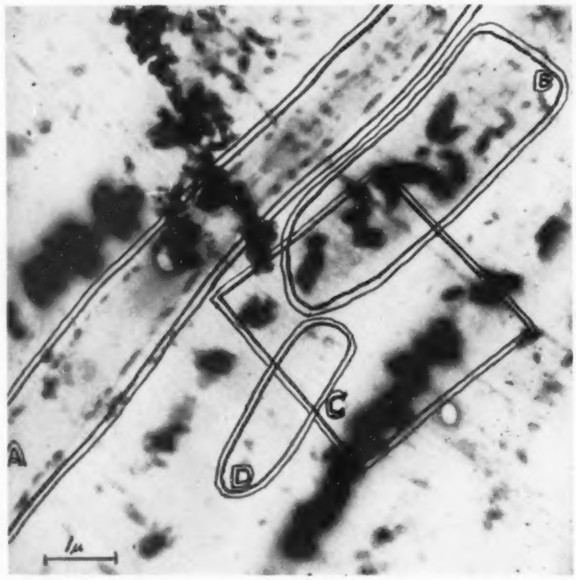


FIGURE 5—Lithium-calcium grease, methacrylate section. Area A—Fiber. Area B—Fiber. Area C—Higher magnification shown in Figure 6. Area D—Small particles between fibers.

Figure 9 shows a section cut from an epoxy resin embedding. This resin has been used to some extent for embedding biologic materials. Experimental embeddings were made with this material to test its sectioning properties and to get a comparison of results between epoxy and methacrylate embeddings. The embedded material in this case was not stained with osmium. The epoxy material is very brittle and is much more difficult to section with a steel knife than is the methacrylate. However, sections cut from the epoxy embeddings show structures comparable to those found in the methacrylate embeddings. For example, the fiber cross sections shown in this figure have a repeated or periodic internal structure and irregular edges.

FIGURE 6—Lithium-calcium grease, methacrylate section. Area A—Build-up of small particles indicating fiber. Area B—Concentration of small particles and fibrils adjacent to larger particles and fibers. Area C—Small fiber like one shown in Figure 4.

Careful examination of these micrographs suggests that the thickener particles are more complex than has been proposed. For example, Vold *et al.*[12], *Vold*[11], and Bondi *et al.*[1] speak of lath-like structures based primarily on X-ray diffraction evidence. This may be true of the building block with which we are dealing. However, these building blocks may be put together in different ways to form various types of thickener particles. For example, in our laboratory two batches of grease of similar formula and identical manufacturing procedures have shown identical X-ray diffraction patterns but different electron diffraction patterns and different thickener particle morphology as shown by the electron microscope. This would indicate identical building blocks or unit cells but different gross structures dependent upon the conditions of composition and procedure under which the final thickener particles were built.

The thickener particle in the lithium-calcium grease under consideration, for example, is a fiber of variable length and cross section, as shown in Figure 3. Its surface is not uniform as shown in Figure 3 and by the edge discontinuities in Figures 4, 5, 6 and 7. These facts suggest the possibility of a mechanism of thickener particle formation as follows: first, nucleation, second, nuclei growth based on the principles of mass action and statistical chance; and third, agglomeration of the particles grown in the second step into the final thickener particles, probably under the influence of shear orientation. Such a mechanism does not exclude the possibility of one type of final thickener particle existing in two or more different simultaneous forms as has been suggested for proteins by Peller[8]. Further evidence is given under the discussion of the results found with the calcium-lead complex thickener, a much more complicated system.

4. *Calcium-Lead Complex Grease*

Figure 10 shows an electron micrograph of a conventional preparation of the thickener found in the complex grease used for this investigation. The method of preparation was the same as that used for the lithium-calcium grease. Several types of thickener particle are shown in the micrograph. These types can be characterized as follows:

1. Filaments with a high length to diameter ratio (the word diameter being used with some misgiving).
2. Bundles of these filaments.
3. Rods and groups of rods.
4. Particles of varying size with rather regular outline but widely varying shape.

This mixture is decidedly different from the lithium-calcium grease where only one general form, a fiber, was found.

The section shown in Figure 11 contains in Area C holes which are lined up in the typical pattern produced by an imperfection in the ultramicrotome knife. The lighter areas between the holes show the scratches produced by the imperfection. Two other areas in this section are important to our study. Area B shows a group of related stained particles indicating a cross section through a non-uniform structure. This may be a particle or rod. Area A shows a filament-particle network.

Area A in Figure 12 shows another filament-particle network of the type shown in Figure 11. These two sections give an indication that one of the functions of the filament structures is to form a network between other types of particles.

Figure 13 shows a section which contains what appears to be "amorphous soap". This area is enlarged in Figure 14. At this magnification it can be seen that this area is not completely devoid of organization. For example, at A there is an edge similar to a particle edge. The areas labeled B contain actual particles. These areas and points are seen in the soap which is called amorphous only because it is of very small size and lack of obvious crystallinity. Further resolution may show even more detailed organization. This section should be compared with Figure 15.

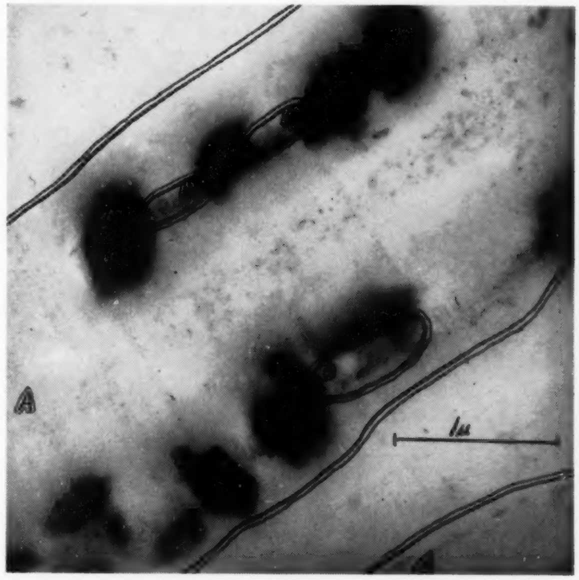


FIGURE 7—Lithium-calcium grease, methacrylate section. Area A—Coordinated system, possibly a fiber. Area B—Small particles associated with larger particles or fibers.

Figure 15 shows a section of a particle with definite but complex organization. The center shows an amorphous character similar to that shown in Figures 13 and 14. This may indicate that the particle formed so rapidly that the amorphous center had no chance

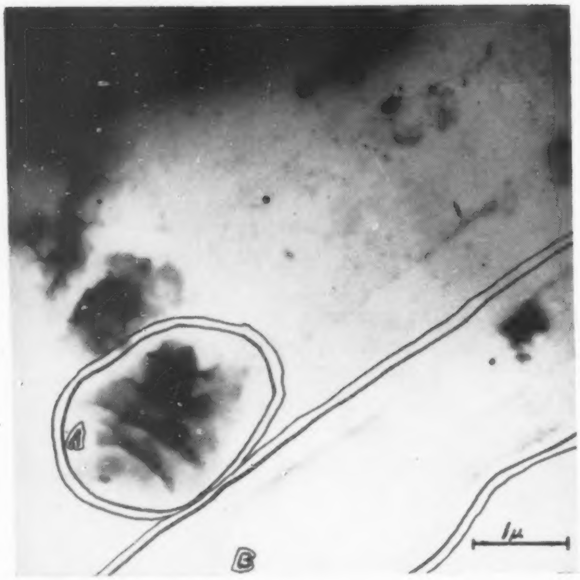


FIGURE 8—Lithium-calcium grease, methacrylate section. Area A—Repeated or periodic structure. Area B—Fiber.

to organize before being removed from the needed environment by the organized surface. The particle is not homogeneous but shows areas of varying density. There are also lines of sharp demarcation between areas of different contrast, indicating that at some time in its history this particle was formed from several other particles. Projections from the side of the particle show to some extent a regular periodic spacing. This regular spacing recalls the condition noted in the

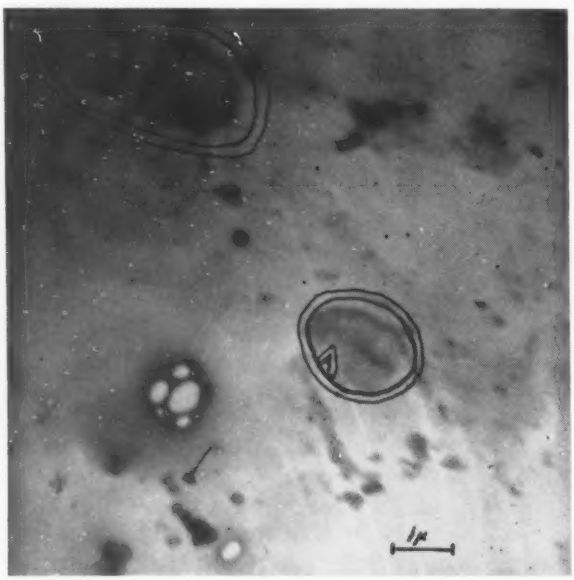


FIGURE 9—Lithium-calcium grease. Area A—Repeated or periodic structure. No stain. Epoxy section.

internal structure of the lithium-calcium grease and suggests that some periodic function may be partially governing particle formation in a lubricating grease thickener.

Figure 16 shows a very small section cut at a time when the angle of the knife with respect to the methacrylate block was changed. This was necessary in order to use a new section of the knife edge. The section cut through three well-defined particles. Two of these appear to be compound, that is, formed from two or more distinct particles. All three particles show some internal structure and in at least one, there seems to be some periodicity to this internal structure. The jet black dots and hair-like areas result from an unfortunate frilling of the emulsion on the photographic plate during development.

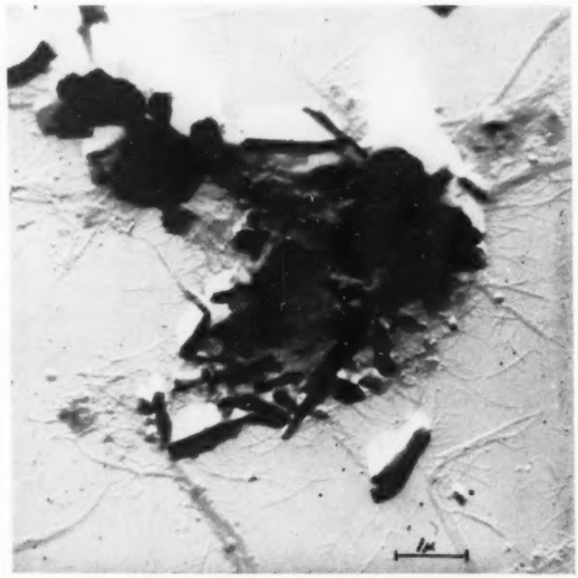


FIGURE 10—Calcium-lead complex grease. Conventional preparation from P.E. suspension, chromium shadowed at ca 20°.

Figure 17 shows a section of a particle organization of considerable complexity. Dense (heavily stained) particles are enclosed in a matrix of considerably less but variable density. Further work needs to be done before sections of this type can be analyzed.

Figure 18 is a section through an area containing principally filaments and bundles of filaments. Particles are also present and there is again a suggestion that the filaments, when available, form a network between particles.

In all of the above discussion it should be borne in mind that, where particles are spoken of, we may actually be dealing with cross sections of the rod structures pointed out in Figure 10. We have no way at present of differentiating between the two.

FIGURE 11—Calcium-lead complex grease, methacrylate section. Area A—Filament-particle network. Area B—Related materials—possible fiber. Area C—Holes resulting from knife imperfection.

The examination of the electron micrographs indicates that the method of thickener particle formation can be the same as that discussed under the lithium-calcium grease; namely nucleation, followed by particle growth and finally particle combination. However, in the case of the calcium-lead complex thickener, we are faced with a complicating situation. In step two, it is apparently possible for the building blocks formed in nucleation to grow in four different ways to form four different types of thickener particle. Possibly this should be reduced to three, if we consider the filaments and bundles of filaments simply variations of the same fundamental method of particle growth. This leaves a considerable number of combinations possible in the final step. The results of the sectioning indicate that many of these combinations are not only possible but actual. For example, Figures 11, 12 and 13 show the association of filaments with particles and Figures 15 and 16 show the association of particles with particles. The elucidation of the mechanism by which these combinations take place lies in the future following the development of further experimental evidence using the techniques of electron microscopy, thin sectioning, serial sectioning, X-ray diffraction and electron diffraction. The reconstruction of the three-dimensional structure from serial sections would be a very important part of the thin sectioning and electron microscope investigations.

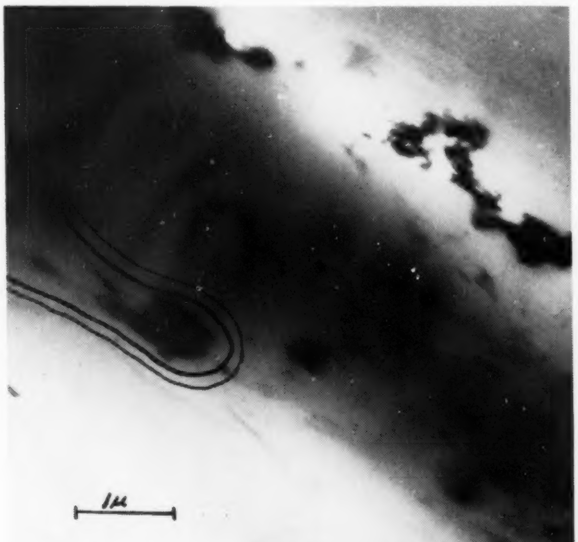


FIGURE 12—Calcium-lead complex grease, methacrylate section. Area A—Filament-particle network.

Acknowledgment

The author wishes to thank Mr. E. L. Armstrong and Dr. David B. Cox of the Socony Mobil research department for many helpful discussions during this investigation and Dr. Emil Borysko of Ethicon, Inc. for a demonstration of his embedding techniques.

Bibliography

1. Bondi, A. *et al.*, Proc. Third World Petroleum Congress, Hague, Sect. 7, 373 (1951)
2. Borysko, E. and Sapranauskas, P., Bull. Johns Hopkins Hosp. *95*, 68 (1954)
3. Borysko, E., Proc. Fourth International Conference on Electron Microscopy, Vol. 2, 40 (1958)
4. Borysko, E. and Roslansky, J., Ann. N. Y. Acad. Sci. *78*, Art. 2, 432 (1959)
5. Farrington, B. B. and Birdsall, D. H., *NLGI Spokesman*, *11*, No. 1, 4 (1947)
6. Farrington, B. B., Ann. N. Y. Acad. Sci. *53*, Art. 4, 979 (1951)
7. McClellan, A. L. and Cortes, J., Jr., *NLGI Spokesman 20*, No. 6, 12 (1956)
8. Peller, L., *J. Phys. Chem. 63*, 1194 (1959)
9. Sjostrand, F. S., *J. Cell. Comp. Physiol. 42*, 16 (1953)
10. Vold, R. D., Coffee, H. F., and Baker, R. F., *NLGI Spokesman 15*, No. 10, 8 (1952)
11. Vold, M. J., *NLGI Spokesman 16*, No. 8, 5 (1952)
12. Vold, M. J., Elersich, V. A., Baker, R. F., and Vold, R. D., *NLGI Spokesman 18*, No. 5, 8 (1954)

Appendix

Procedure and Apparatus

Sample Preparation for Conventional Electron Micrographs

The method of sample preparation of anhydrous calcium acetate complex type thickeners can drastically affect the structure observed. In particular, any washing of the soap structures with petroleum ether after drying changes their form. However, washing is desirable to remove the oil so that shadowing of the

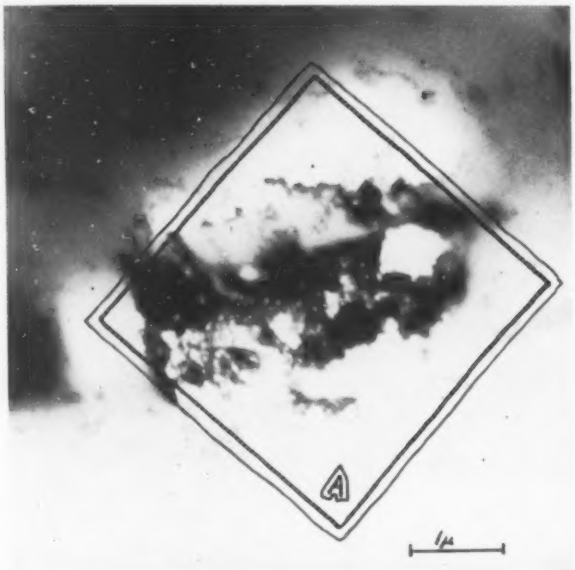


FIGURE 13—Calcium-lead complex grease, methacrylate section. Area A—Amorphous soap—Area shown at higher magnification in Figure 14.

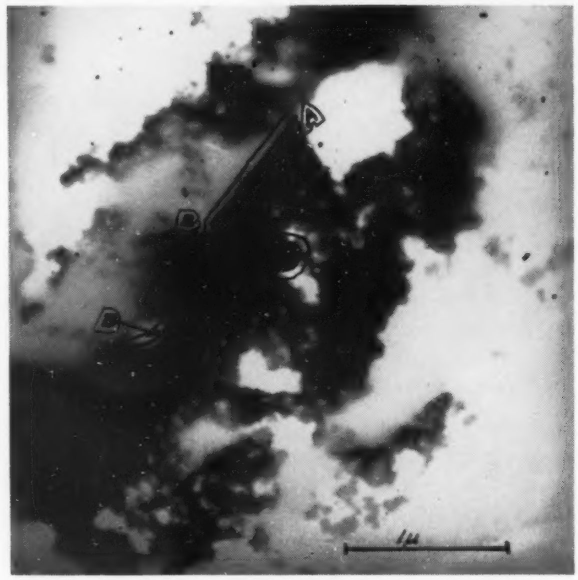


FIGURE 14—Calcium-lead complex grease, methacrylate section. Area A—Particle edge. Area B—Particle.

thickener particles rather than of the oil will be obtained. To overcome this problem, a new method of washing was developed which uses air drying only as the final step.

A small sample of the grease to be inspected (about the size of a pea) is uniformly suspended in about 10 ml. of petroleum ether in a 15-ml conical bottom centrifuge tube. The tube is then spun in an oil testing centrifuge at 1500 rpm for five minutes. Most of the liquid is then sucked out of the tube, leaving 0.5-1.0 ml. of liquid above the thickener in the bottom of the tube. Petroleum ether is then added to bring the volume again to about 10 ml. and the thickener resuspended by stirring. A motor-driven wire stirrer is a useful aid in obtaining rapid, uniform redispersion. The centrifuging, liquid removal and resuspension are repeated a second time. A drop of this final suspension is then pipetted from a capillary to an electron microscope grid previously coated with Formvar resin using conventional methods. The drop is allowed to evaporate. The specimen so prepared is shadowed at an angle of 15-20° with chromium and examined in the electron microscope.

This method was used in preparing the samples of lithium-calcium and calcium-lead complex greases for the conventional electron micrographs shown in this paper.

Sample Preparation for Sectioning

A sample of the grease to be examined was spread in a thin layer on an acid-cleaned microscope slide using a blood smear technique. A second acid-cleaned slide was used as the spreading tool. About one-half of the slide was covered with the sample. This technique undoubtedly could result in some working of the sample and consequent thickener particle alignment. However, the smearing is accomplished in one pass so the effect should be minimized. Also, the edge of the slide used for smearing is sufficiently rough to provide areas where very little working takes place.

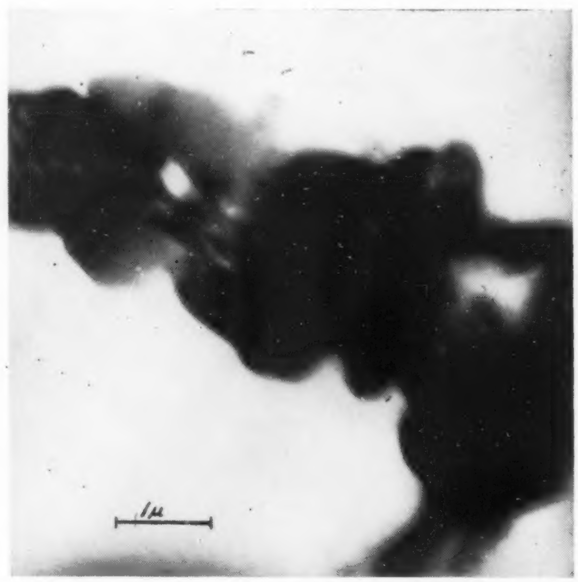


FIGURE 15—Calcium-lead complex grease, methacrylate section. Irregularly-shaped compound particle with apparent amorphous center.

The slides containing the smeared grease were then placed in a Coplin jar containing petroleum ether. Leaching of the oil was very rapid, being essentially complete in one minute. The slides were then transferred to a second Coplin jar and then to a third, each containing fresh petroleum ether. The coated half of the slide was kept at the bottom of the jar and transfer was made as quickly as possible to prevent air drying followed by rewetting. The slide was removed from the final petroleum ether bath and air-dried in a petri dish. If the films on the slide are kept sufficiently thin, there is no tendency of either the grease or the thickener particles to slough off in the Coplin jars.

A small ball of cotton or filter paper was then placed in the petri dish alongside the slides containing the

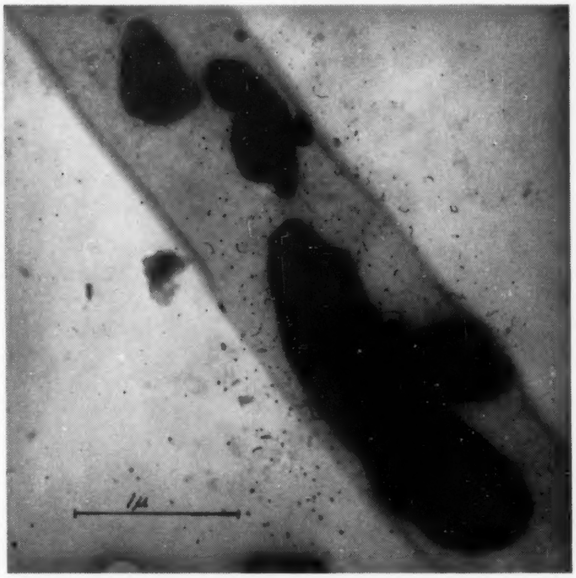


FIGURE 16—Calcium-lead complex grease, methacrylate section. Particles with regular outline in matrix of fine particles.

washed smear of thickener. The petri dishes were then placed in an oven and preheated to 40-50°C. The dishes were then removed from the oven, 10-20 drops of a 1 per cent solution of OsO_4 were placed on the cotton, and the dishes were returned to the oven. They were allowed to remain in the oven until fixing and staining of the smeared thickener by osmium vapor was completed (approximately one-half hour). This operation changes the color of the thickener from white to light brown. The samples were then ready for embedding. This fixing with osmium allows the complex thickeners to be rewet without damage.

It would be possible for a change in thickener particle morphology to take place during osmium staining. To make sure that this did not occur a conventional

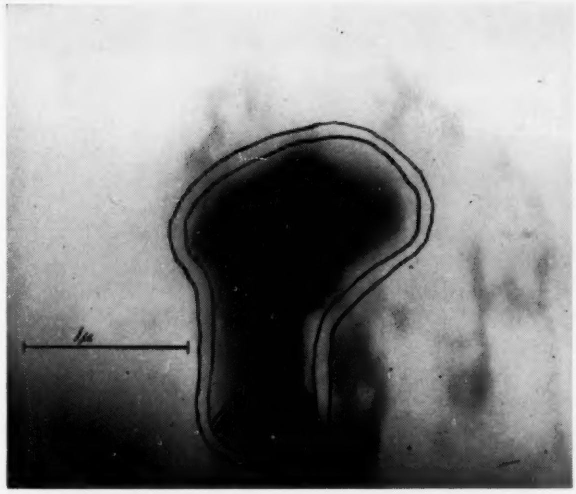


FIGURE 17—Calcium-lead complex grease. Area A—Dense particles buried in matrix of methacrylate section.

preparation of the lithium-calcium grease was first stained with osmium as described above. Electron micrographs of such stained specimens are shown in Figures 19A and 19B. The morphology shows the same fibrous character as that shown in Figures 3A, 3B and 3C. However, we see some internal structure in the fibers as a result of the variable osmium absorption rather than the surface structure seen in the shadowed specimens. As a final indication that no change in morphology takes place during staining, stained conventionally-prepared specimens of the lithium-calcium grease were shadowed with chromium. Electron micrographs of these specimens are shown in Figures 20A

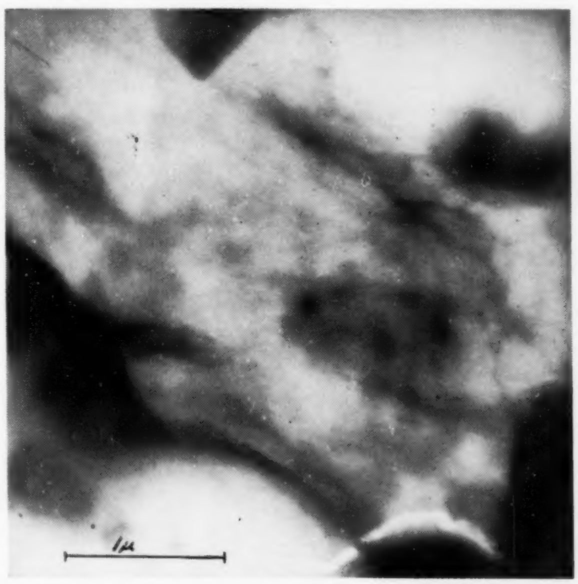


FIGURE 18—Calcium-lead complex grease. Thin fibrils of methacrylate section.

and 20B and comparison with Figures 3A, 3B and 3C indicates identical type structures in the stained and in the unstained specimens.

Embedding

The embedding procedure was that of Borysko and Roslansky[4] and Borysko[3]. The microscope slide holding the stained smears of lubricating grease thickener was placed, smear side up, in the center of a 2-inch x 4-inch rectangle of aluminum foil. The foil was then folded up around the edges of the microscope slide to form a shallow tray with walls extending above the surface of the slide.

Methyl methacrylate and butyl methacrylate monomers were washed free of inhibitors and dried. A 1:1 mixture of these monomers containing 1 per cent of benzoyl peroxide was used for embedding. Some of this mixture was partially polymerized to a high viscosity in a drawn out glass culture tube using the method described by Borysko and Sapranauskas[2]. This partial polymerization was performed in a water bath maintained at 70-80°C. The tubes were removed frequently and shaken so that the resulting polymer was a uniform liquid of high viscosity. When the desired viscosity was obtained, the tubes were removed from the water bath and placed in an ice bath.

Sufficient monomer to just cover the specimen was then placed in the aluminum foil tray or boat containing the stained smear on the microscope slide. This step prevents bubble formation between the glass slide and the aluminum. (The monomer is evaporated during the heating cycle used for final polymerization.) The tip was broken off one of the tubes containing the partially polymerized methacrylates, the tubes being first carefully dried to prevent water from dripping onto the specimen. The material was then forced out of the tube by air pressure (rubber bulb) into the boat to a depth of about 3 mm. Any persistent bubbles were removed by touching the area above them with a drop of the monomer solution or by teasing them out with a needle. The aluminum boat was then placed in an oven at 50-55°C. The temperature was raised in uniform steps of 5-8°C. to a final temperature of 90-110°C over a period of about 3 hours. The specimens were left in the oven overnight at this top temperature to complete the polymerization. The next morning the samples were removed from the oven and cooled to room temperature. The aluminum foil was stripped from the specimens and the methacrylate wafer, containing the stained thickener, separated from the glass slide by placing the slide in contact with a block of dry ice. The rough edges of the wafer were smoothed with a file. These wafers were then sectioned. Examination by phase microscopy and electron microscopy at each stage of the preparation and embedding process has indicated no particle damage. Extended storage (one week) of the grease in the monomer did not change the particle morphology.

Sectioning

A piece approximately 0.5 x 1.0 cm. was cut from the polymer wafer and mounted in the specimen holder of a Sjostrand ultramicrotome[6]. This specimen holder was modified by including a Timken test block of tool steel to act as one jaw of a vise for the specimen. The set screw acted to tighten the vise and the normal face of the specimen holder acted as the other jaw of the

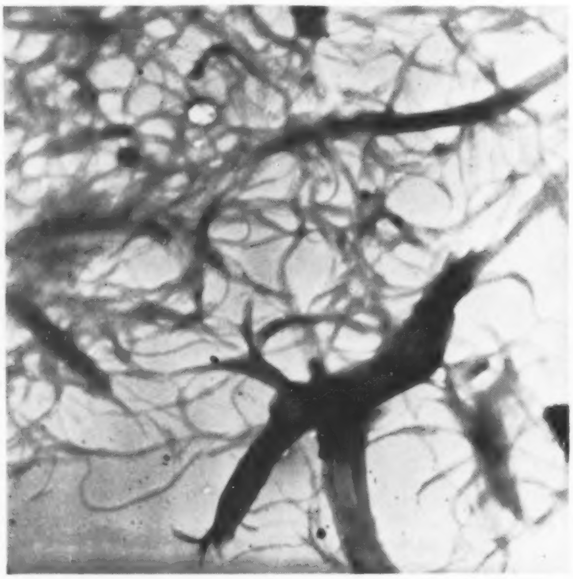


FIGURE 19A—Lithium-calcium grease. Conventional preparation from P.E. suspension, osmium stained.

FIGURE 19B—Lithium-calcium grease. Conventional preparation from P.E. suspension, osmium stained.

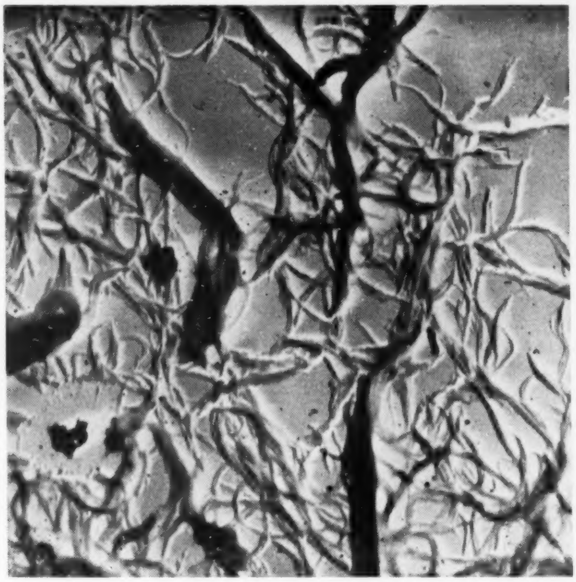


FIGURE 20A—Lithium-calcium grease. Conventional preparation from P.E. suspension, osmium stained, then chromium shadowed at ca 20°.

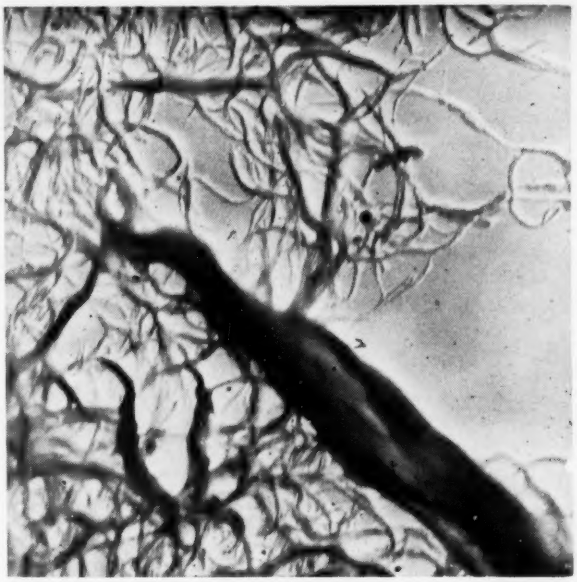


FIGURE 20B—Lithium-calcium grease. Conventional preparation from P.E. suspension, osmium stained, then chromium shadowed at ca 20°.

vise. The specimen was adjusted to project 3-4 mm. from the front of the specimen holder.

The front face of the specimen was trimmed away to leave a small projection in the form of a four-sided truncated pyramid. The truncated face was approximately 0.5 by 0.5 mm. and the pyramid was about 0.5 mm. high. The trimming was done free-hand with a microtome blade under a low power microscope. It could be done more conveniently and accurately with a micromanipulator.

The specimen holder was then mounted on the microtome and the truncated face of the pyramid brought to a final plane surface using the intermittent motor drive of the microtome and manual advance of the knife. Sections were then cut from the specimen using the continuous motor drive and the thermal advance of the specimen against the knife in fixed and locked position. The sections were collected on distilled water or on 20 per cent ethyl alcohol in distilled water in the unmodified trough of the ultramicrotome. These sections were on the order of 100-150Å thick. Because of the large area of the trough, serial sections tended to separate and could not be obtained in the desired ribbon form. Restriction of the volume of the collecting trough may overcome this problem. The sections were picked up on electron microscope grids coated with Formvar resin and examined in the electron microscope.

Electron Microscope

The electron microscope used for these studies was a modified RCA Model EMU-2A. It is equipped with an RCA intermediate lens and modified stage. This instrument uses a Canalco lens power supply, externally centerable condenser aperture and pole piece compensator with externally centerable objective aperture. All the electron micrographs shown in this paper were taken with a 10-mil condenser aperture and a 3-mil objective aperture. ■

About the Author

J. W. Wilson, Jr. *received an AB degree from Amherst college in 1938 and studied at M.I.T. the following year. He started his career with Socony Mobil Oil Co., Inc. in 1934 as a service station attendant. While still attending school, he worked in the research department at the Brooklyn laboratory as a summer employee from 1936 to 1939. In 1940 he joined the staff as an analytical chemist. He is presently a senior technologist in the applied research and development division of the Paulsboro, N. J., laboratory. Mr. Wilson is a member of the American Chemical Society and the Electron Microscope Society of America. His published work has dealt with the color of petroleum products, microscopic studies of the structure of lubricating greases and special techniques for use in electron microscopy.*

Literature and Patent Abstracts

Ingredients

Aluminum Soap Hydrocarbon Thickener with Increased Solvation

Aluminum ethyl hexoate soaps are rendered more soluble in hydrocarbons by the addition of a small amount of coconut oil fatty acids to the precipitated soap prior to filtering and drying it. The amount of acids used depends upon the content of free aluminum hydroxide. Leverberg and Baumgartner make this suggestion in U.S. Patent 2,978,-411, assigned to Esso Research and Engineering Co.

Such a soap was cited which contained 1.7 per cent excess aluminum hydroxide and which took 20 minutes to gel gasoline so that no settling would occur. When 0.8 weight per cent of coconut oil fatty acids was added before filtering and washing, the aluminum hydroxide content was 0.9 per cent and the gel time was 10 minutes.

Application

Multi-Purpose Lubricants

Lubrication Engineering 17, pp. 160-4, 1961. Commentary by Sargent and Belt.

While the discussion deals primarily with fluid lubricants, the mention of lubricating greases indicates that our industry is more advanced than the suppliers of the fluid type. Lubricating greases are dismissed with this statement: "Multi-purpose greases, thanks to the new lithium and calcium complex bases, are already in wide use and more than 90% of the grease requirements of almost any operation can be satisfied with a single grease."

Additives

Lead Soaps Prepared from Petroleum Oxidates

According to Christensen in U.S. Patent 2,978,470, assigned to Tex-

aco Inc., oil soluble lead soaps comparable to lead naphthenate can be made from the oxidation products of certain lubricating oils. First, the oils used should contain little wax. Next, the saponifiable portion of the oxidate is extracted from the material before the soap is formed.

An illustration is given of a case where the starting material was an Edeleanu refined and acid treated distillate fraction from a naphthenic base crude. This had a viscosity of 99.6 SUS at 100°F. The oxidation was carried out until the saponification number was 102 after which the desirable fraction was naphtha extracted before converting first to sodium soap and then to lead soap. This soap had a solubility of 9.05 per cent in paraffin oil and gave a Mean Hertz Load of 31.

Composition

Lubricating Greases Thickened with Soap-Salt Complexes

Morway in U.S. Patent 2,976,-242, assigned to Esso Research and Engineering Co., describes lubricating greases in which the bodying agents are formed by reacting hydrated lime with a mixture of acetic acid, a medium molecular weight carboxylic acid containing 3 to 10 carbon atoms, and a high molecular weight fatty acid. The medium weight acids may be straight or branched chain, hydroxy or aromatic. The mole ratio of the acetic acid used should be at least 4:1 and that of the medium to the high molecular weight acids in the range of 0.5:1 to 10:1.

Such a lubricant is illustrated by one made from 12 per cent glacial acetic acid, 3 per cent Hydrofol acids 51, 3 per cent benzoic acid, 9.7 per cent hydrated lime, 0.5 per cent phenyl alpha-naphthylamine and 71.8 per cent oil of 55 SUS at 210°F. The oil, Hydrofol acids, lime, and benzoic acid were mixed and heated to 135°F before the acetic acid was added. The charge was then heated to 510°F, cooled to 250°F, where the inhibitor was added, further cooled to 180°F and passed through a Gaulin homogenizer.

The finished lubricating grease had a worked penetration of 208 which changed to 250 after 100,000 strokes in a grease worker. The dropping point was 500+ and tested on a Timken machine, the product carried a load of 50 pounds. Also, the lubricant was insoluble in boiling water and lost five pounds

in an oxygen bomb test in 365 hours.

Soap-Salt Complexes as Lubricating Grease Thickeners

Calcium soap-salt complexes, made by reacting a mixture of acetic acid, high molecular weight fatty acids and a hydrolyzation product of lactams with hydrated lime, are used as thickeners in lubricating greases and described by Morway and Matuszak in U. S. Patent 2,976,-241, assigned to Esso Research and Engineering Co. The mole ratio of the acetic acid to the other acids is preferably from 0.6:1 to 6:1 and the mole ratio of the amino-carboxylic acid to the high molecular weight acid is from 2:1 to 10:1.

For example, a lubricating grease was prepared from the following weight per cents of ingredients: glacial acetic acid 12, epsilon-caprolactam monomer 4, stearic acid 2, hydrated lime 10, phenyl alphanaphthylamine 0.5 and lubricating oil of 55 SUS at 210°F 71.5. The oil and lime were mixed to a smooth slurry in a fire-heated kettle. The caprolactam was then added and the mixture heated for two hours at 150°F before the stearic acid and the acetic acid were added. Heating was then continued to about 500°F over a period of two hours. The mass was then cooled to 200°F where the inhibitor was added. After further cooling to room temperature, the batch was passed through a Gaulin homogenizer at 5000 psi.

The product was a smooth grease with a worked penetration of 340, dropping point of 500+, insoluble in boiling water and stable for 400+ hours in a bomb oxidation test. The lubricant carried 15 weights in an Almen Test and lubricated a bearing operating at 10,000 rpm and 250°F for over 1000 hours.

Stable Calcium Acetate-Mineral Dispersions

Simple dispersions of calcium acetate in lubricating oil can be stabilized against settling by use of a small quantity of certain amines. Morway and Smith, who describe

such compositions in U.S. Patent 2,976,243, assigned to Esso Research and Engineering Co., find tertiary alkyl amines and particularly Primene JM-T satisfactory.

While the examples of the compositions are fluids, it is stated that lubricating greases can also be formed with the suggested mixtures. One formulation included 25 per cent of glacial acetic acid, 17 per cent of hydrated lime, and 2 per cent of amine. The latter material was added with the lime before the introduction of the acetic acid.

Lubricating Grease Thickeners Containing Salts of Organic-Substituted Phosphonic Acid

Thickeners consisting of complexes of salts of certain phosphonic acids and either salts of low molecular weight acids or soaps of high molecular weight fatty acids, are useful for bodying lubricating fluids. Such combinations are described by Morway, Bartlett and Muessig in U.S. Patent 2,977,303, assigned to Esso Research and Engineering Co. The phosphonic acids used are 1-hydroxy-alkyl or alkenyl wherein such groups contain ten to 30 carbon atoms. Either alkali or alkaline earth metals can be used to form the complexes.

For example, a lubricating grease was made from the following all in parts by weight: 22 of mustard seed oil, 4.95 of sodium hydroxide, 1 of sodium sulfonate, 70.55 of lubricating oil having a viscosity of 55 SUS at 210°F, 2.5 of 1-hydroxy-1-methylhexadecyl phosphonic acid and 1 of phenyl alpha naphthylamine. The mustard seed oil, mineral oil and sulfonate were mixed and warmed to 150°F before the sodium hydroxide was added as a forty per cent solution. Upon continued heating the mass ceased to foam at about 350°F but started again at about 450°F. Heating was continued to 520°F where the temperature was maintained for 30 minutes, until foaming ceased. After heating was discontinued and the temperature dropped to 300°F, the phosphonic acid was added and the mixture stirred for 30 minutes before the addition of the oxidation inhibitor. Finally at 150°F the product was passed through a Gaulin homogenizer at 6000 psi.

This gave a lubricant with a worked penetration of 250 which changed to 290 after 100,000 strokes. The dropping point was 500+ and it operated in a 204 bearing at 10,000 rpm for 30 minutes with a temperature rise of 75°F.

Composition and Processing

Producing Heat-Stable Lubricating Greases

Bergen et. al. in U.S. Patent 2,977,300 (assigned to Continental Oil Co.) describe the production of lubricating greases in which the thickening agent consists of colloidal calcium carbonate coupled with certain organic acidic materials. The latter acids include itaconic, pyromellitic, furoic, terephthalic, etc. The calcium carbonate is first formed and then acid equivalent to 37 to 85 per cent of that required for neutralization of the calcium compound is added.

Preparation of the lubricant entails five steps as follows:

(a) Forming an alcoholic solution of calcium sulfhydrate by passing hydrogen sulfide through an alcoholic slurry of calcium hydroxide;

(b) Admixing the resulting alcoholic slurry of calcium sulfhydrate with an oil soluble alkyl aryl sulfonic acid dissolved in a natural or synthetic oil;

(c) Precipitating from the latter solution a stable colloidal dispersion of oil insoluble calcium carbonate in particles less than 0.25 micron diameter by passing carbon dioxide through the solution.

(d) Mixing the calcium carbonate dispersion with an acid as indicated above and in the ratio indicated.

CLIMAX MOLYBDENUM

Molysulfide® News Digest

CLIMAX MOLYBDENUM COMPANY, a division of American Metal Climax, Inc., 1270 Avenue of the Americas, New York 20, N.Y.

RECENT TEST SHOWS MORE PROOF OF MoS_2's LOAD-CARRYING ABILITY

Grease Compounders Take Hard Look At Paste Type Concentrates

In a recent issue of NLGI SPOKESMAN, a prominent lubrication authority warns that in the future an increasing number of the newer machines will be greased "for life"—or for long lubrication intervals. He also says that as the industrial pace quickens and wages go up, it will be more economical to use more expensive grease and reduce lubrication frequency.

That's why grease compounders and blenders are looking into every possible way to maintain and increase their profit levels.

High on investigation priority lists are the paste type concentrates. These concentrates contain solid lubricants. Molysulfide concentrates can be applied by grease guns or by brushing, and will stand up at temperatures as low as -100 F. and peaks as high as +500 F.

Concentrate users include a fast spreading number of industries. Major manufacturers in the automotive industry use paste type Molysulfide concentrates for pre-assembly lubrication of splines, cams, etc.

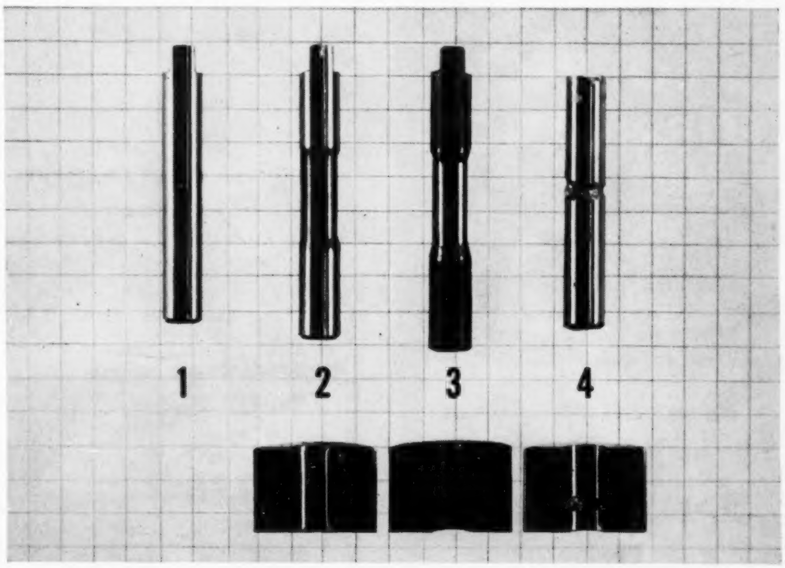
The aviation and missile industry is using an increasing amount to overcome low-temperature problems in servo-mechanisms, bearings and gears.

General industry is applying them, not only on metals exposed to continuous high temperatures, but also on plastic bearings, machine tool ways, press fittings and other moving parts where long lubricant life and high load carrying capacity is needed.

Much remains to be learned about the full potentialities of paste type concentrates but all signs indicate they are one of the answers to lubrication problems of the future.

When writing, refer to CL-110

From the German laboratory of Alpha Molykote Corporation comes new proof of Molysulfide's ability to resist galling and seizing at pressures beyond the yield point of most metals. Previous tests have shown that Molysulfide provides positive protection up to 475,000 psi. In this Alman Wieland test, similar to the Falex test, a ¼-inch pin actually extruded *without any surface damage* at pressures of 100,000 psi, while those using other lubricants were torn, galled or "frozen" to the point of breakage.



Photograph above shows mild steel test pins. No. 1: unused pin. No. 2 (lubricated .with mineral oil and Molysulfide) and No. 3 (with Molysulfide bonded coating) were subjected to rotating pressures between bearing halves. Both were elongated and extruded without galling, seizing, or weight loss. No. 4 shows typical failure with conventional lubricant. Note that the key sheared off and pin and block were galled and seized.

Extensive surveys among a great many independent laboratories indicate that the extrusion phenomenon produced with Molysulfide has never been achieved with any other conventional lubricant.

This proven load carrying ability of Molysulfide is only one of the reasons why grease compounders and lubrication engineers are investigating and testing new uses for MoS_2.

Other reasons include its extremely low coefficient of friction; its wide temperature range from -300 F to 750 F; its tenacious adherence to metal surfaces and a resultant great resistance to "scrape-off" and "wash-off"; and, its high chemical stability combined with long-life characteristics.

The uses for solid lubricants are multiplying. And—by test—Molysulfide is the superior solid lubricant.

When writing, refer to CL-111

(e) Agitating and removing the volatile solvents from the mixture.

For example, 3000 grams of methanol, 192 grams of calcium oxide, and 125 grams of Hyflo were added to a 5-liter flask which was then placed in an ice bath. The mixture was blown with hydrogen sulfide while agitated. The temperature was not permitted to rise above 46°C during this step and the reaction was considered complete when the temperature fell to 6°C after which the product was filtered.

Next, 2927 grams of postdodecylbenzene sulfonic acid in naphtha, 3000 ml. of benzene and 1641 grams of the calcium sulfhydrate in methanol, which was prepared in the first step, were mixed and blown with carbon dioxide until essentially all of the hydrogen sulfide had been expelled. After the addition of 1608 grams of hexa(2-ethyl butoxy) disiloxane, there remained a colloidal suspension of about 8.4 per cent calcium carbonate.

To a mixture of 206 grams of this dispersion, 181 grams of hexa (2-ethyl butoxy) sisiloxane and 200 ml. of benzene, was added a mixture consisting of 19.4 grams of itaconic acid, 0.5 ml. of water, 20 ml. of tert butyl alcohol, 25 ml. of methanol, 100 ml. of acetone and 200 ml. of benzene. The total mixture was heated to reflux the solvents for 45 minutes before they were removed by heating to 292°F under reduced pressure. After a final heating to 356°F in the presence of dry nitrogen a translucent, brown lubricating grease of 300 penetration resulted.

While the above patent covers the method of production of this type of lubricating greases, U. S. 2,977,301 by the same authors, covers the compositions wherein the modified colloidal calcium carbonate is employed.

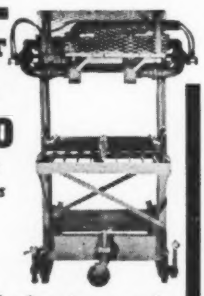
Composition

Lubricating Grease Useful for Food Processing and Canning Machinery

Worth in U.S. Patent 2,978,410, assigned to Union Oil Co. of California, describes lubricating greases for use in the presence of water, steam and corrosive fruit juices, which also protect the equipment against corrosion. Such a product consists of a lubricating oil thickened with 5 to 15 per cent of calcium 12-hydroxystearate soap and also containing 1.5 to 4 per cent of a metal oxide, and 0.75 to 2 per cent of an anti-corrosion agent. The metal oxides used are those of zinc or magnesium or mixtures of the two in which the zinc oxide predominates. An oxidaton inhibitor and Paratac are also included in the composition. The anti-corrosion agent consists of 1-hydroxyethyl-2-heptadecenyl glyoxalidine marketed under the trade name Amine 220.

The soap is formed in the presence of a portion of the oil at a temperature below 280°F and after addition of further oil and cooling the other ingredients are added, with the Paratac introduced when the mass is about room temperature.

For example, a lubricating grease was made with the following



weight per cents: 6.6 12-hydroxystearic acid; 1.0 hydrated lime; 2.1 zinc oxide; 0.4 magnesium oxide; 0.4 Paratac; 0.5 Agerite Stalite; 1.1 Amine 220; and 87.9 300 neutral oil. The product had a worked penetration of 290 and softened 8 points upon working 100,000 strokes. The dropping point was 293°F and the pressure drop four pounds in 100 hours in an oxidation test.

Plug Valve Lubricant and Sealant

Compositions, which are relatively resistant to aliphatic hydro-carbons, mineral and vegetable oils, natural and manufactured gases, oils and gases containing hydrogen sulfide or mercaptans, water, dilute aqueous solutions of mineral acids and alkalies up to a pH of 13, are described by Scherer and Bierwagen in U.S. Patent 2,979,457, assigned to Rockwell Manufacturing Co. Such mixtures consist essentially of 75 to 95 parts by weight of a synthetic liquid organic polysulfide polymer having an average molecular weight of from 1000 to 4000 (such as Thiokol LP-32), 2 to 7 parts of fine silica and 0.1 to 1.5 parts of polytetrafluoroethylene resin. Up to 10 parts of either chlorinated biphenyl or mixed isomeric terphenyls may also be included.

Thus, 89.7 parts of polysulfide polymer, 5 parts of chlorinated biphenyl and 2 parts of fine mica were mixed and heated to 250°F. As the mass cooled 2.6 parts of fine silica were added and when the temperature dropped to 120°F, 0.7 parts of an aqueous dispersion of 60 per cent polytetrafluoroethylene resin were added. After reheating to 250°F, a product suitable for use over a temperature range of 0° to 200°F was obtained.

Testing

The Effect of Radiation and Other Environmental Conditions on the Performance of Greases
***Lubrication Engineering* 17, p. 188 (1961) by Agius and Morris.**

The published material is an abstract of a complete paper and hence is limited in information. Apparently carbon black, Bentone, lithium soaps and both calcium and sodium complex soap thickeners were used in the products tested. Esters are mentioned as the base fluid.

In addition to exposure to radiation, the samples were exposed to carbon dioxide first in bombs and next in a ball bearing rig operating at elevated temperatures. The bearing under a carbon dioxide atmosphere was subjected to a 100 pound thrust load and operated at 1440 rpm for 100 hours. At 150°C the lithium base product disintegrated and bled badly, whereas the two non-soap products were in excellent condition. The calcium complex lubricant was intermediate in condition. At 200°C a sodium complex thickened lubricating grease broke down whereas the carbon black and Bentone thickened lubricants were in good condition.

NLGI Associate, Technical

CONTAINER AND CLOSURE MANUFACTURERS

American Can Company
4810 Belleview, Kansas City 12, Mo.
Representative—H. T. Rich

American Flange & Manufacturing Company, Inc.
30 Rockefeller Plaza, New York 20, N. Y.
Representative—Richard L. Parish, Jr.

Bennett Industries
Peotone, Illinois
Representative—S. A. Bennett

Central Can Company, Inc.
3200 South Kilbourn Ave., Chicago 23, Ill.
Representative—Henry Frazin

Cleveland Container Company
4919 So. Halstead St., Chicago 9, Ill.
Representative—R. D. Sayles

Continental Can Company, Inc.
633 Third Ave., New York 17, N. Y.
Representative—W. J. Flint

Geuder, Paeschke & Frey Company
324 North Fifteenth St., Milwaukee 1, Wis.
Representative—Neil Savee

Greif Brothers Cooperage Corp.
1821 University Ave., St. Paul 4, Minn.
Representative—Ray Suttle

Inland Steel Container Company
6532 South Menard Ave., Chicago 38, Ill.
Representative—J. Daniel Ray

Jones & Laughlin Steel Corporation
Container Division
3 Gateway Center, Pittsburgh 30, Pa.
Representative—J. E. Morris

National Steel Container Corp.
6700 South LeClaire Ave., Chicago 38, Ill.
Representative—Henry Rudy

The Ohio Corrugating Company
917 Roanoke Ave. S. E., Warren, Ohio
Representative—Lawrence F. McKay

R. C. Can Company
9430 Page Ave., St. Louis 32, Mo.
Representative—Harry H. Ellerbrock

Republic Steel Corporation
Container Division
465 Walnut Street, Niles, Ohio
Representative—Theodore Humphrey

Rheem Manufacturing Company
400 Park Ave., New York 22, N. Y.
Representative—G. Gwyn Tucker

Rieke Metal Products Corporation
Auburn, Indiana
Representative—Raymond F. Ouer

Sefton Fibre Can Company
Div. Container Corp. of America
3275 Big Bend Blvd., St. Louis, Mo.
Representative—W. V. Swofford

Southline Metal Products Company
3777 West 12th Street, Houston 24, Tex.
Representative—Warren F. Wackman

Steel Package Division of National Lead Company
722 Chestnut Street, St. Louis 1, Mo.
Representative—Warren T. Trask

Thatcher Glass Mfg. Co., Inc.
Plastic Container Division
375 Park Ave., New York 22, N. Y.
Representative—Henry E. Griffith

United States Steel Products
Division, United States Steel Corporation
30 Rockefeller Plaza, New York 20, N. Y.
Representative—C. R. Justice

Vulcan-Associated Container Companies, Inc.
P. O. Box 161, Bellwood, Ill.
Representative—V. I. McCarthy, Jr.

ENGINEERING SERVICES

The C. W. Nofsinger Company
307 East 63rd Street, Kansas City 13, Mo.
Representative—C. W. Nofsinger

Sumner Sollitt Co.
307 N. Michigan Ave., Chicago 1, Ill.
Representative—A. J. Barth

MANUFACTURERS OF EQUIPMENT FOR APPLICATION OF LUBRICATING GREASES

Balcrank, Inc.
Disney near Marburg, Cincinnati 9, Ohio
Representative—Richard P. Field

The Farval Division Eaton Manufacturing Co.
3249 East 80th St., Cleveland, Ohio
Representative—E. J. Gesdorf

Gray Company, Inc.
60 Northeast 11th Ave., Minneapolis 13, Minn.
Representative—B. A. Beaver

Lincoln Engineering Division McNeil Machine & Engineering Co.
4010 Goodfellow Ave., St. Louis 20, Mo.
Representative—R. E. Crean

Stewart-Warner Corporation
Alemite Division
1826 Diversey Parkway, Chicago 14, Ill.
Representative—E. G. Wicklatz

MARKETING ORGANIZATIONS

Ampol Petroleum, Ltd.
Buchanan Street
Balmain, New South Wales, Australia
Representative—M. E. Brownell

California-Texas Oil Company
380 Madison Ave., New York 17, N. Y.
Representative—Hal U. Fisher

Canadian Petrofina Limited
505 Dorchester Boulevard West
Montreal, Quebec, Canada
Representative—M. E. Wight

Cooperative GLF Exchange, Inc.
Terrace Hill, Ithaca, N. Y.
Representative—W. S. Miller

Denco Petroleum Company
5115 Denison Avenue, Cleveland 2, Ohio
Representative—E. E. Busse

Derby Refining Co.
420 West Douglas, Wichita, Kan.
Representative—W. B. Neil

D-X Sunray Oil Company
Mid-Continent Bldg., P. O. Box 381, Tulsa, Okla.
Representative—J. W. Basore

Farmer's Union Central Exch., Inc.
P. O. Box G, St. Paul 1, Minn.
Representative—H. F. Wagner

Illinois Farm Supply Company
P. O. Box 585, Bloomington, Ill.
Representative—S. F. Graham

Lubrication Engineers, Inc.
P. O. Box 7128, Fort Worth 11, Texas
Representative—James P. Bell

M.F.A. Oil Company
P. O. Box 510, Columbia, Mo.
Representative—Stuart L. Spradling

Pennsylvania Refining Company
2686 Lisbon Road, Cleveland 4, Ohio
Representative—Ben Sollitto

United Co-Operatives, Inc.
111 Glamorgan, Alliance, Ohio
Representative—A. J. Miller

and Marketing Members

Valvoline Oil Company
Division of Ashland Oil & Refining Co.
Box G, Freedom, Penna.
Representative—R. L. Sailer

REFINERS

Henry H. Cross Company
600 South Michigan Ave., Chicago 5, Ill.
Representative—J. N. Waddell

SUPPLIERS OF EQUIPMENT FOR MANUFACTURING LUBRICATING GREASES

Barrett Manufacturing Company
P. O. Box 8096, Houston 4, Texas
Representative—George J. Barrett, Jr.

Chemicolloid Laboratories, Inc.
55 Herricks Road, Garden City Park, N. Y.
Representative—David F. O'Keefe

Girdler Process Equipment Division, Chemetron Corp.
2820 West Broadway, Louisville, Ky.
Representative—J. E. Slaughter, Jr.

Stratford Engineering Corporation
612 W. 47th Street, Kansas City 12, Mo.
Representative—D. H. Putney

Struthers Wells Corp.
1003 Pennsylvania Ave. West, Warren, Pa.
Representative—K. G. Timm

SUPPLIERS OF MATERIALS FOR MANUFACTURING LUBRICATING GREASES

American Potash & Chemical Corp.
99 Park Avenue, New York 16, N. Y.
Representative—W. F. O'Brien

Archer-Daniels-Midland Company
Industrial Chemicals Division
P. O. Box 532, Minneapolis 40, Minn.
Representative—J. H. Kane

The Baker Castor Oil Company
Bayonne, N. J.
Representative—J. W. Hayes

Baroid Chemicals, Inc.
A subsidiary of National Lead Company
1809 South Coast Life Building
Houston 2, Texas
Representative—C. M. Finlayson

Cabot Corporation
141 W. Jackson Blvd., Chicago, Ill.
Representative—Warren M. Parsons

Climax Molybdenum Company
1270 Avenue of the Americas, New York 20, N. Y.
Representative—K. B. Wood, Jr.

Darling & Company
4201 South Ashland Ave., Chicago 9, Ill.
Representative—L. Strauf

E. I. du Pont de Nemours & Company, Inc.
Wilmington 98, Delaware
Representative—Joseph J. Mikita

The Elco Lubricant Corporation
Jennings Road & Denison Avenue
Cleveland 9, Ohio
Representative—R. K. Smith

Emery Industries, Inc.
4300 Carew Tower, Cincinnati 2, Ohio
Representative—David R. Eagleson

Enjay Chemical Company
Div., Humble Oil & Refining Co.
15 West 51st St., New York 19, N. Y.
Representative—Stephen L. Wythe

Foote Mineral Company
18 W. Chelten Ave., Philadelphia 44, Penna.
Representative—W. M. Raynor

A. Gross and Company
295 Madison Avenue, New York 17, N. Y.
Representative—Eugene W. Adams

Harchem Division Wallace & Tiernan, Inc.
25 Main St., Belleville, N. J.
Representative—H. M. Abbott

Humko—Chemical Department
P. O. Box 398, Memphis 7, Tenn.
Representative—W. J. O'Connell

Lithium Corporation of America, Inc.
500 Fifth Ave., New York 36, N. Y.
Representative—Malcolm M. Moore

The Lubrizol Corporation
Box 3057—Euclid Station, Cleveland 17, Ohio
Representative—J. B. Irwin

Mallinckrodt Chemical Works
2nd & Mallinckrodt Sts., St. Louis 7, Mo.
Representative—John A. Caughlin

Monsanto Chemical Company
800 North Lindbergh Blvd., St. Louis 66, Mo.
Representative—J. W. Newcombe

Newridge Chemical Company
7025 West 66th Place, Chicago 38, Ill.
Representative—T. E. Shine

M. W. Parsons—Plymouth, Inc.
59 Beekman St., New York City 38, N. Y.
Representative—Herbert Bye

Werner G. Smith, Inc.
1730 Train Avenue, Cleveland 13, Ohio
Representative—W. Meckes, Jr.

Swift & Company
165th & Indianapolis Blvd., Hammond, Ind.
Representative—F. H. Beneker

Synthetic Products Company
1636 Wayside Rd., Cleveland 12, Ohio
Representative—Garry B. Curtiss

TECHNICAL AND RESEARCH ORGANIZATIONS

Battelle Memorial Institute
505 King Avenue, Columbus 1, Ohio
Representative—R. L. Jentgen

The Chek-Chart Corporation
222 West Adams St., Chicago 6, Ill.
Representative—H. Eldridge

Chemico (Pty.) Ltd.
Miller and 7th Streets, P. O. Box 6349,
New Doornfontein, Johannesburg,
South Africa
Representative—O. Richter

Compagnie Francaise de Raffinage Direction Recherches et Procedes
22, Rue Boileau, Paris (16e) France
Representative—Albert E. Miller

Institut Francais du Petrole
CMR—Courtel, 4 Place Bir Hackeim
Rueil—Malmaison (S. et Oise) France

LABOFINA S. A.
Centre de Recherches du Groupe PETROFINA
Bruxelles 12, Belgium
Representative—R. Gillerot

Phoenix Chemical Laboratory, Inc.
3953 Shakespeare Ave., Chicago 47, Ill.
Representative—Mrs. G. A. Krawetz

People in the Industry

Sonneborn Chemical and Refining Names New Board Chairman and President

The election of Rudolf G. Sonneborn as chairman of the board of Sonneborn Chemical and Refining corporation and William Wishnick as president and chief executive officer has been announced by Witco Chemical Company, Inc. Sonneborn is a wholly owned subsidiary of Witco and constitutes its largest manufacturing division.

Mr. Sonneborn was formerly president of the Sonneborn company. Both he and Mr. Wishnick continue as Witco directors. Mr. Wishnick also continues as Witco's executive vice president.

Their offices are located at Sonneborn's executive headquarters in New York City.

Sonneborn is a major producer of white oil, petrolatum and petroleum sulfonates. It manufactures and markets Pennsylvania-grade motor oils and lubricants under the "Amalie" trade name.

Other Witco divisions manufacture and market a broad line of chemicals and allied products including detergents and detergent chemicals, phthalic anhydride, urethane chemicals, carbon black, metallic soaps, and tar and asphalt products.

Dixon Names Zimmer Senior Vice-President

The Joseph Dixon Crucible Co., Jersey City, N. J., has announced the promotion of Warren A. Zimmer to senior vice-president. He had formerly been executive vice-president.

In other promotions H. J. Aderente was advanced from assistant comptroller to comptroller, and Walter H. Buckhout was named chief financial officer as well as treasurer.

ADM Appoints Two Regional Sales Managers

Appointment of two regional sales managers for Archer-Daniels-Midland Co.'s chemical group was announced by Hall S. Dillon, group sales manager.

They are Anton Dorfmueller Jr., Cleveland, Ohio, manager for the east central and middle Atlantic states, and Robert C. Fulton, Minneapolis, manager of the midwest region.

Dorfmueller joined ADM in 1955 as head of foundry products sales in the Buffalo, N. Y., area. Since then he has advanced through several sales positions in the company's Federal Foundry Supply division to his present post as division manager. He will continue in the latter capacity in addition to his new duties as regional sales manag-

er for all chemical group products. Dorfmueller has been associated with the foundry industry since his graduation from the University of Wisconsin in 1948.

Fulton started with ADM's Alfalfa division in 1953 in Neodesha, Kans. In 1958 he was appointed manager of that division, with headquarters in Kansas City, Mo. Fulton was named director of production and administration of ADM's former specialty group in 1959 and early this year was assigned special duties within the chemical group. He attended Westminster college and the University of Missouri.

Appointment of Dorfmueller and Fulton completes the new regional sales organization for the ADM chemical group. Previously, Everett Sklarz, New York, was named manager of the eastern region and Robert W. Mairs, Los Angeles, western regional manager.

Appoints Harry Carroll

Harry L. Carroll has been appointed district sales manager for American Potash & Chemical Corp., effective July 1, it was announced by W. W. Young, southern regional sales manager.

Carroll, who assumes responsibility for marketing industrial chemicals in the southeastern area, will continue to make his headquarters in the company's Atlanta regional sales office.

C. L. Straughan continues as district manager in charge of potash and agricultural chemical sales.

Completes Addition

American Flange & Manufacturing Company has announced the completion of a new factory addition and enlarged facilities at 1100 West Blancke St., Linden, N. J.

This expansion was necessary to accommodate growing pail closure and plastic manufacturing operations and afford better utilization of Tri-Sure closure service facilities at Linden.

The Chicago plant at Kilpatrick Ave. and Arlington St., will serve Tri-Sure customers geographically situated for best service as before.

Industry News

Publishes Catalog

Lincoln Engineering Co. of St. Louis, has published a new 32-page catalog on air-powered equipment for materials dispensing applications.

Equipment shown in Catalog 42 includes pumping systems to dispense paint, cold roofing materials, food materials, caulking compounds and sealers, mastic adhesives and glues, vinyl material for plastic molding, inks, underbody coatings, sound deadeners and similar fluid or semifluid materials.

Specifications are given on Lincoln "Power-Master" pumps, dispensing accessory units, measuring valves and airless hydraulic "Dyna-Spray" units and their accessories. Also listed are hose, air controls, couplers, nipples and adapters needed for systems. The catalog contains many photos showing the equipment in use.

For a free copy write: Lincoln Engineering Co., 4010 Goodfellow Blvd., St. Louis 20, Mo.

Bonded Dry-Film Lubricant

An improved bonded dry-film lubricant for cutting tools is available from the Alpha-Molykote Corp. Known as Molykote PVE-K250 the new coating can be applied to any clean metal surface. It replaces Molykote PVE, which could only be applied to low alloy ferrous metals. In addition, the new compound is a single package coating, in contrast to its predecessor which was a two-package system requiring mixing before application.

Molykote PVE-K250 lengthens the life of such tools as gear cutters, reamers and taps by preventing metal pick-up or build-up on cutting tool flanks. It is easily applied by spraying and requires no surface preparation other than cleaning. As it is non-inflammable, no spray booth is necessary.

After tools have been evenly sprayed with a thin coat of the new product and allowed to dry in air for 30 minutes, the coating is cured for one hour in an oven heated to 140°F. Parts are ready to use after cooling.

Additional information about Molykote PVE-K250 may be obtained without cost from the Alpha-Molykote Corp., 65 Harvard Ave., Stamford, Conn.